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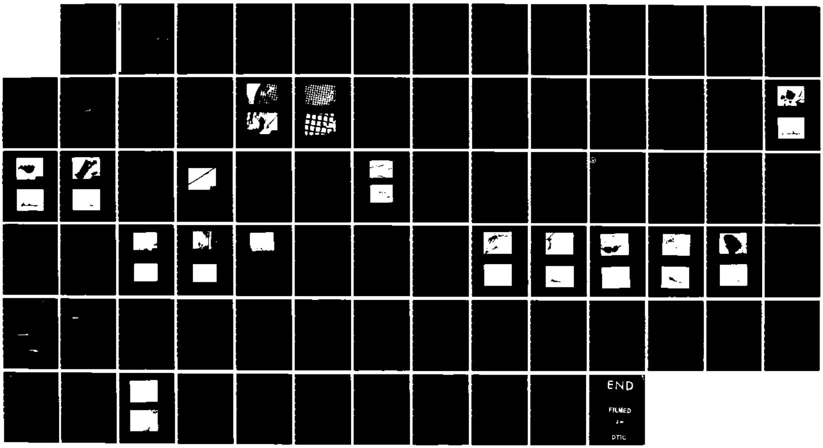

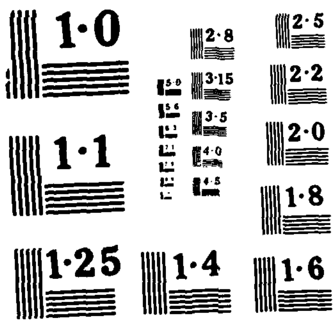

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UNUSUAL CONTAMINANT IN JP-4 FUEL DELIVERED TO FORT CAMPBELL, FORT KNOX, AND TENNESSEE ARMY NATIONAL GUARD

AD-A163 399

INTERIM REPORT
BFLRF No. 201

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By

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Under Contract to

U.S. Army Belvoir Research and Development Center
Materials, Fuels and Lubricants Laboratory
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Many common types of debris were encountered during the course of a recent investigation to identify an unusual contaminant present in jet fuel (JP-4) at three Army locations. Most of the other debris were materials commonly found in fuel samples, e.g. metal flakes, rust, fibers, dirt, etc. Various analytical techniques used in this evaluation indicated that the unusual flake-like organic debris was probably a manmade (synthetic) organic component and not a fuel product. It had a high melting point (>200°C) and was insoluble in most common solvents. The physical appearance of the flakes (thin and flat while floating in the fluid) indicated that the material was originally formed or deposited in a thin layer prior to its introduction into the fuel.

The most likely source of a material with these characteristics was an unpigmented paint or protective coating from one of the epoxy or similar resin families. Such a material (cont'd)

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could be introduced into the fuel by debonding or delamination of the contaminant from any handling, storage, or transportation equipment that contains such a coating. The elements, chemical bonds, and functional groups detected by the various analytical tests were consistent with this conclusion.

The use of a light box for enhanced visual ("Clean and Bright") evaluation is presented in this report. Also discussed is an approach for isolating similar debris (from fuel) to provide for scanning electron microscopic, Auger, and Electron Spectroscopy for chemical analysis (ESCA) evaluation.

FOREWORD

This report was prepared at the Belvoir Fuels and Lubricants Research Facility (SwRI), Southwest Research Institute, under DOD Contract No. DAAK70-85-C-0007. The project was administered by the Fuels and Lubricants Division, Materials, Fuels, and Lubricants Laboratory, U.S. Army Belvoir Research and Development Center, Fort Belvoir, Virginia 22060-5606, with Mr. F.W. Schaekel, STRBE-VF, serving as Contracting Officer's representative; and with Messrs. M.E. LePera and W.R. Williams serving as contracting officer's technical representatives.

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Southwest Research Institute funded the improved visual contaminant viewing procedure and the contaminant isolation technique which are contained within this report. The helpful assistance of Messrs. Moody, Marable, and Hansard of Fort Campbell Army Air Field and Col. Otis, Army Aircraft Maintenance, 5th Transportation BN, Fort Campbell Army Air Field, is gratefully acknowledged.

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I. INTRODUCTION AND BACKGROUND

The U.S. Army Belvoir Research and Development Center was notified of a JP-4 fuel contamination problem by the Tennessee Army National Guard in March 1985. Preliminary definition of the problem and recommendations as to necessary follow-up actions for disposal of the contaminated fuel supplies were initiated and are covered in a letter report provided as Appendix A to this report. Subsequently, on April 11, 1985, five 1-gallon samples (all-level) of stored JP-4 were received at the Belvoir Fuels and Lubricants Research Facility (SwRI), BFLRF,* from the Tennessee Army National Guard (Smyrna, TN) for detailed analysis to identify fiber-like contaminant and other particulate debris as to its origin. Debris was found to be present, including:

- Type 1; Small black particles (soft), high chlorine content
- Type 2; Small black particles (hard), possibly solder fragments
- Type 3; Small milky white flakes, possibly man-made nonwoven fabric fragments
- Type 4; Clumps of lint-like fibers, adhered to a yellowish, waxy-appearing substance, probably lint and paper fibers
- Type 5; Small metallic-looking flakes of relatively pure aluminum

Fuel visual appearance showed white flakes and fibers. The report on these analyses is provided in Appendix B.

On 23 April 1985, three 1-gallon samples of JP-4 arrived at BFLRF for analysis to identify "very minute white-looking particles resembling dandruff" based on a letter from Headquarters, U.S. Army Armor Center and Fort Knox (dated 16 April 1985) in which Lewis N. Simpson (petroleum foreman) stated:

> "Per instructions from USAGMPA, New Cumberland Army Depot, this sample of JP-4 is being sent to you for your disposition."

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* Formerly U.S. Army Fuels and Lubricants Research Laboratory (AFLRL). AFLRL redesignated as BFLRF on 1 October 1985.

Information pertaining thereto is as follows:

1. Tanker arrived at Fort Knox, KY at approximately 1110 hr 12 Apr 85. Accompanied by Mr. Hendricks, DFSC, Cameron Station, VA and a QAR representative from Louisville, KY.

2. We used clean sample containers. They were thoroughly rinsed with the product on board by opening manifold outlet line. They were rinsed three (3) times. Then we filled the sampler from same outlet for observation.

3. We observed a few very, very minute white-looking particles resembling dandruff. We had to look very, very hard and close to detect them. But we all did see them.

4. We then took these three (3) one (1) gallon samples from compartment #2 by using a bacon bomb. They represent a bottom, middle and top sample. The cans are thus marked."

Analytical findings regarding these samples are contained within this report.

On 9 April 1985, BFLRF received a memorandum for record from the Belvoir Research and Development Center (STRBE-VF) dated 3 April 1985 summarizing "JP-4 Fuel Contamination Problem and User Assistance by BRDC" which is provided in Appendix C. On 28 April, BFLRF was directed to visit Fort Campbell Army Air Field regarding the quality of JP-4 fuel used in aircraft. A trip report covering the period 1-7 May 1985 technical liaison visit to Fort Campbell is provided in Appendix D.

After having received a number of tank truck deliveries of JP-4 highly contaminated with the unusual flake material, "white suspended matter and excess fiber," personnel at Fort Campbell Army Air Field began rejecting their fuel deliveries in March-April 1985. The rejection was based on the fuel specification workmanship requirements in MIL-T-5624 that fuel be clean and bright, i.e., completely free from visually-observed haze, fibers, or particles, even though the fuel met all other requirements. Alternative fuel sources were then used, and no further fuel was delivered to Fort Campbell, Fort Knox, or Smyrna (ANG) from commercial sources until an acceptance guarantee could be made and the source of the contamination could be identified. During this same period (March-April), an UH-60 helicopter experienced a flame-out at Fort Campbell. Inspection of the fuel filter by Fort Campbell personnel showed it to be covered with a lint-type material. Actions were then taken to replace the fuel filters with new elements on all UH-60's at Fort Campbell. Analyses of the lint by Fort Campbell sources indicated that it was principally composed of cellulose (cotton). In May, all UH-60's were grounded for other reasons. In late April 1985, one Air Force F-16 crashed less than 5 minutes after takeoff from the Fort Campbell Army Air Field. The F-16 had refueled from Tank No. 202, which still contained some of the contaminated fuel. However, no connection between the fuel quality and the crash were ever established. Filter elements from the filter separator for Tank No. 202 were subsequently replaced with new elements because the pressure differential had reached 19.5 psig (max allowable is 20 psig). As reported in Appendix D, most of the "dirtiest" JP-4 was delivered to Fort Campbell Army Air Field fuel receiving Tank No. 308, which still contained some fuel having the "white flakes." Laboratory personnel at Fort Campbell Army Air Field provided assistance in evaluating filter separator efficiency, visual identification of particles of greatest concern ("white flakes") in the fuel, and waxy looking flakes viewed on screen test filters under a low-power microscope. Based on activities at Fort Campbell during the liaison visit, it was concluded:

1. Presence of unusual "dandruff-like flakes" in fuel at Fort Campbell Army Air Field was confirmed, at least in Tank No. 308, which received most of the "worst-appearing" fuel.

2. No strong evidence was found to substantiate suspicion that "flakes" were passing through filter separators even though (what appeared to be) "flakes" were present in fuel at the Oasis POL Station and in samples from helicopters.

3. Ordinary trash and system debris, as well as unusual "wax flakes," were observed on test filters from the POL tank fuel. Emphasis should be placed on identifying the "wax flakes" rather than the lint, glass fiber, and inorganic debris.

4. The fragile nature of the "wax flakes" could lead to the flakes breaking apart in the filter separators and subsequently reforming through agglomeration in the filtered fuel at a later time (a highly hypothetical suggestion for which no evidence was observed).

This report summarizes testing done to identify the unusual "flakes" observed in JP-4 fuel delivered to Fort Campbell, Fort Knox, and the Tennessee Army National Guard.

II. TECHNIQUE DEVELOPMENT

A listing of samples examined is presented in Table 1. Visual examination of all fuel samples confirmed the presence of flake-like materials in addition to ordinary contaminants. Sample AL-14205-T was selected as the best candidate for detailed analytical testing because it exhibited a greater density of flake concentration. Also the larger flakes present in this sample were more readily removable from the sample in quantities sufficient for analysis. The other samples were also examined by a variety of techniques as described in this report.

A. Enhanced Visual Evaluation

The flakes present in the "as received" samples could be detected by close inspection of the fuel through a clear glass bottle in sunlight, or by passing a beam of light with a microscope illuminator through the side of the bottle. The flakes were, however, difficult to see, and improved means to readily detect their presence visually was obviously needed.

TABLE 1. SAMPLE DESCRIPTION

AFLRL Sample Code Number	Date Received	Description	Received From
AL-14063-T	4-11-85	Tank No. 2, All-Level	Tennessee Army National Guard, Smyrna, TN
AL-14089-T	4-23-85	Tank Truck No. 15136, Top	Fort Knox, KY
AL-14090-T	4-23-85	Tank Truck No. 15136, Middle	Fort Knox, KY
AL-14091-T	4-23-85	Tank Truck No. 15136, Bottom	Fort Knox, KY
AL-14175-T	5-13-85	Tank No. 1, Bldg. 5251, Bottom	Fort Knox, KY
AL-14235-T	5-30-85	Tank Truck No. 117, Composite From Compartment 1, Delivery into Tank No. 345	Fort Knox, KY
AL-14247-T	6-04-85	Tank Truck No. 117, Manifold of Compartment 1, Delivery into Tank No. 345	Fort Knox, KY
AL-14171-T	5-08-85	Tank No. 202, Lower 1/3	Fort Campbell Army Air Field, KY
AL-14205-T	5-21-85	Tank No. 308, Bottom	Fort Campbell Army Air Field, KY
AL-14172-X	5-08-85	UH-1H Fuel Filter Elements, 2 each	Fort Campbell Army Air Field, KY
AL-14173-X	5-08-85	Filter Separator Elements From Filter Unit for Tank No. 202, 2 each	Fort Campbell Army Air Field, KY
AL-14239-X	5-31-85	Filter Separator Elements From Filter Unit for Tank No. 202, 3 each	Fort Campbell Army Air Field, KY
--	5-07-85	UH-60 Fuel Filters, 2 each	Fort Campbell Army Air Field, KY
--	5-07-85	Lint Sample From UH-60 Fuel Filter From Aircraft that Experienced Flame Out	Fort Campbell Army Air Field, KY
--	4-28-85	Filter Element, Outer Sock Samples From Two Different Vendors	U.S. Army Belvoir Research and Development Center, VA

Several lighting techniques were evaluated to optimize visual detection of the flakes. The superior approach involved bright illumination of the sample container from the bottom and the container being viewed from the side with a black background behind the viewing area. Figure 1 shows the lightbox constructed to view the samples. Flakes, fibers, and other debris were readily observable when the sample bottle was swirled to suspend the contaminants in motion and placed in the lightbox. The entire contents of the bottle was readily viewable, and observations could be made concerning relative concentration of materials present, the tendencies to settle, or stay in suspension, and the color, shape, etc., of the materials.

B. Recovery of Contaminants

Initial attempts at recovery of the flake-like material by conventional means, such as filtering the material through ASTM D 2276 type membrane filters, proved to be unacceptable. The major problem encountered was contamination of the unusual flake-like material with other normally encountered filterables in the fuels, such as small quantities of dirt, rust, fibers, etc. To analyze the flakes properly, it was important that they be removed in as clean a condition as possible, avoiding other extraneous materials present in the fuel.

To accomplish this task, approximately 750 mL of the sample fuel were placed in a separatory funnel and allowed to stand for at least 10 minutes. The particles appeared to be slightly more dense than the fuel and therefore tended to settle to the bottom. The bottom portion (5 to 10 mL) of the sample was then decanted off, affording a very high concentration of the heavier contaminants into a small volume of fuel sample. The sample was then poured into a watch glass and observed under a stereo-microscope (40X). The heat of the microscope illuminator produced convection currents in the fuel, and the readily visible flakes tended to "drift" through the fuel, while the heavier materials, such as rust and dirt, settled to the bottom of the watch glass. Small (3-mm diameter) transmission electron microscope (TEM) grids of 200 mesh copper screen held by tweezers were then used to "scoop" the flakes from the fuel.

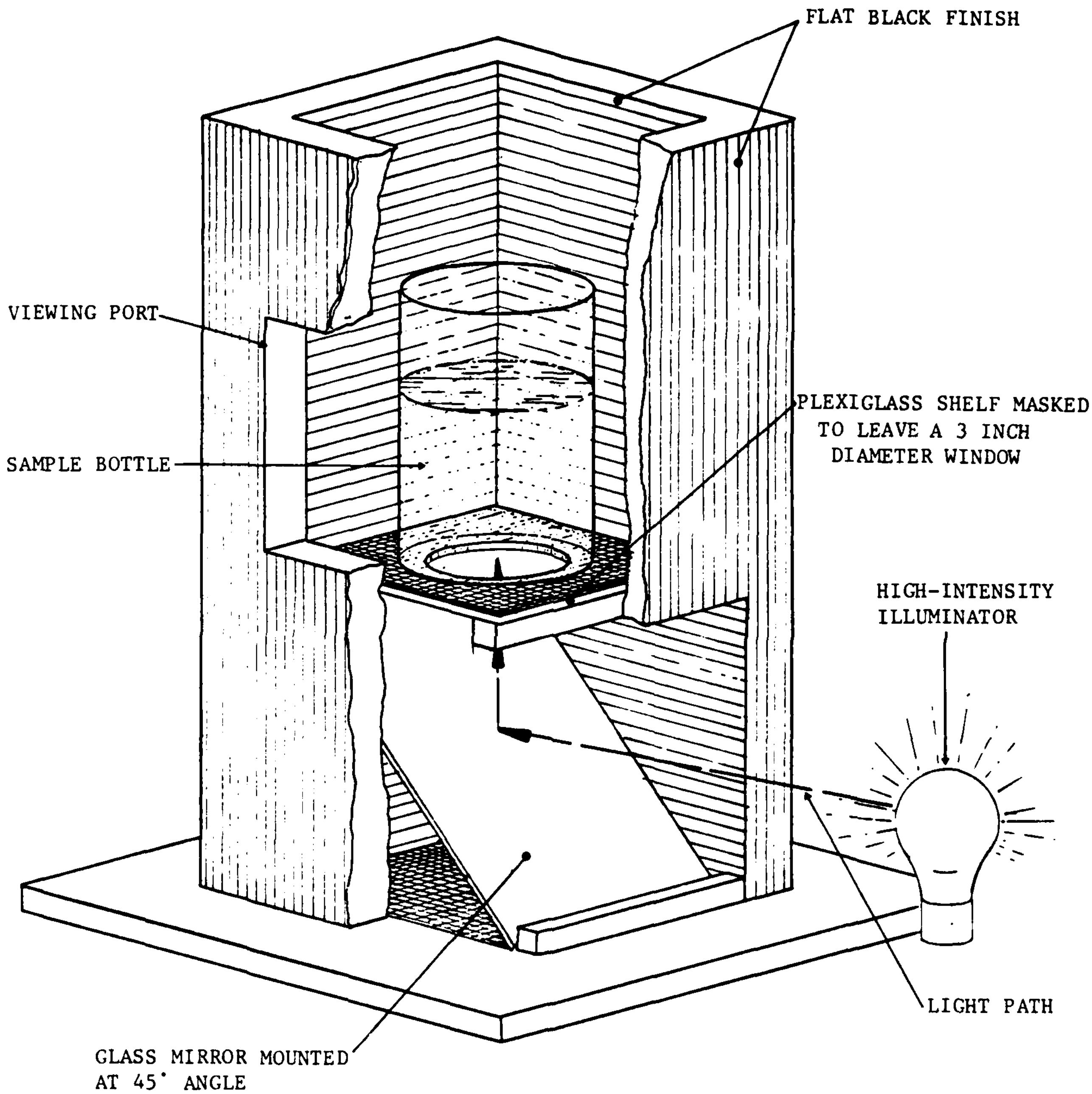


FIGURE 1. LIGHTBOX FOR ENHANCED VISUAL EVALUATION

The bottom side of the grid was then touched lightly with blotting paper to wick away most of the remaining fuel present. The flake on the top of the grid was "washed" to remove any extraneous material from the flake by gently immersing the grid in methanol. The flake was observed through the microscope to ensure that the flake could be recaptured if it should float away from the grid. The grid was then removed from this wash bath, the bottom of the grid was blotted, and the sample allowed to air dry. This procedure was repeated until sufficient material was recovered for the various analytical techniques. The flake-like material was removable in most cases from the TEM grid by manual manipulation of the flake with a fine-pointed needle. Figure 2 illustrates the recovered flakes on TEM grids.

III. ANALYTICAL TESTS OF FUEL SAMPLE AL-14205-T (Fort Campbell Sample From Tank No. 308 Bottom)

A. General Description of Recovered Material

Fuel sample AL 14205-T from the bottom of Tank No. 308 was selected for the most comprehensive analysis because of its large concentration of flake-like material. The flake-like particles were examined by low-power microscope (10 to 40X) while still floating in the fuel. The "dandruff-like" flakes, noted in the field of observation, were observed as thin flat flakes that varied from transparent to milky in color. Some of the flakes had on their surfaces small brown spots which appeared to be dirt or iron oxides embedded in the flake material. The flakes were thin, with an irregular perimeter which, in most cases, approximated a square. The larger flakes varied from 200 to 400 micrometers in size, and were relatively few in number as compared to the smaller flakes of <100 micrometers. While still in the fluid, the flakes were flexible. If a flake was captured with precision tweezers while floating in the fluid, the flake would wrap around the tweezer tip without fragmenting when removed from the fluid. Flakes could also be removed from the fluid by carefully scooping them from the fluid using a small-diameter TEM grid of 200-mesh copper screen as discussed in Section II of this report. Accumulating quantities of material in this manner was an extremely time-consuming task. When allowed to dry on the grid, the flakes would often develop a "mud crack" like pattern and fragment into several smaller pieces. In other cases, the flakes would roll up into a scroll-like shape during drying. Once dried, the material was a light yellow to amber color. Typical flakes from this sample are shown in Figures 2 and 3.

B. Specific Analytical Tests

Solubility of the flakes was tested in a variety of solvent materials, including water, acetone, methyl ethyl ketone, tetrahydrofuran, methanol, dichloromethane, and a triple solvent of toluene, acetone, and methanol. All of these common solvents had no noticeable effect on the flake material.

The flakes could also be heated to as high as 230°C with no evidence of decomposition or melting using microscopic observation.

After usable samples were obtained and washed, they were mounted for scanning electron microscope (SEM) observation and energy-dispersive analysis of X-rays (EDAX) by attaching the copper grids to an aluminum SEM stub. Elemental analysis exhibited a high background in the low energy range with trace amounts of Fe. This high background in the low energy range indicates that the flakes are organic and that small specks of other contaminants have embedded themselves into the flakes. All samples were therefore screened with EDAX to ensure that they were relatively "clean" before being submitted for further analysis by other techniques.

Auger spectrometer analysis was performed on several of the flakes from sample AL-14205-T. These analyses revealed the flakes to be composed primarily of carbon with detectable oxygen and nitrogen, which again suggests that they are organic in nature. Typical Auger spectra are shown in Figure 4. No element common to grease structure (lithium, calcium, etc.), paint pigments (titanium, zinc, chromium, etc.), or common conductivity additives (calcium, chromium, sulfur) was detected.

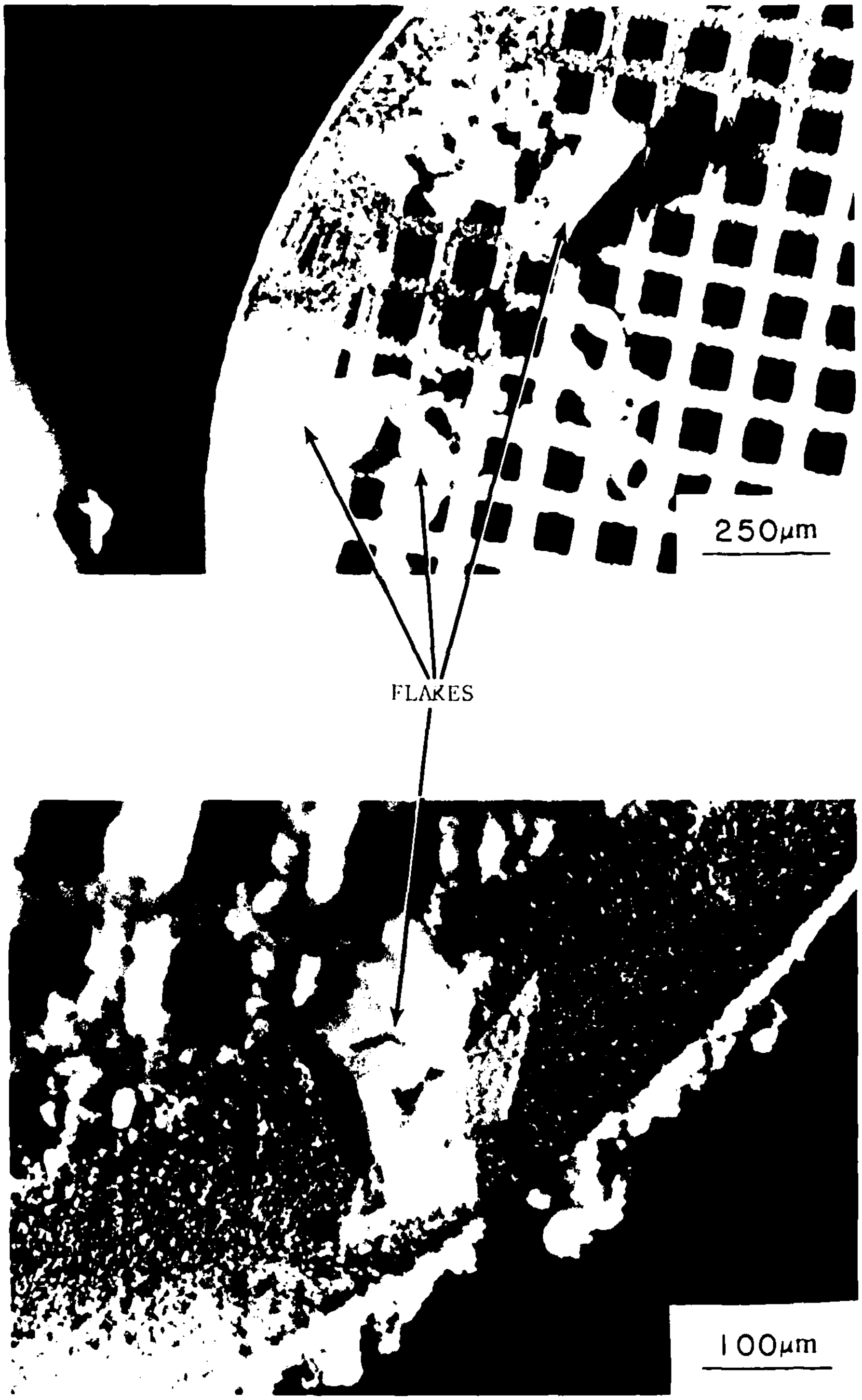


FIGURE 2. RETRIEVED CONTAMINANT FLAKES ON TEM GRID

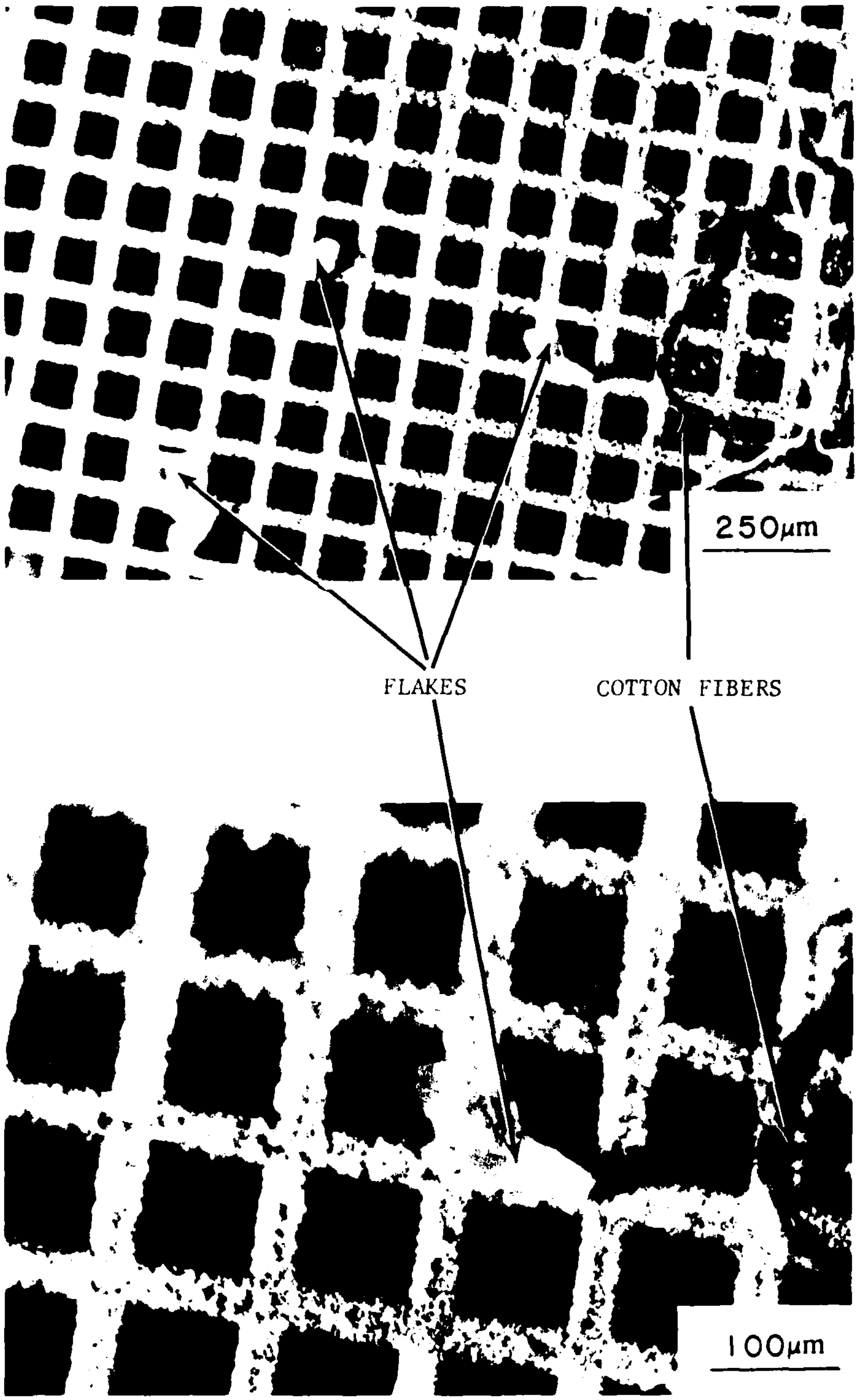


FIGURE 3. TYPICAL FLAKES RETRIEVED FROM SAMPLE AL-14205-T

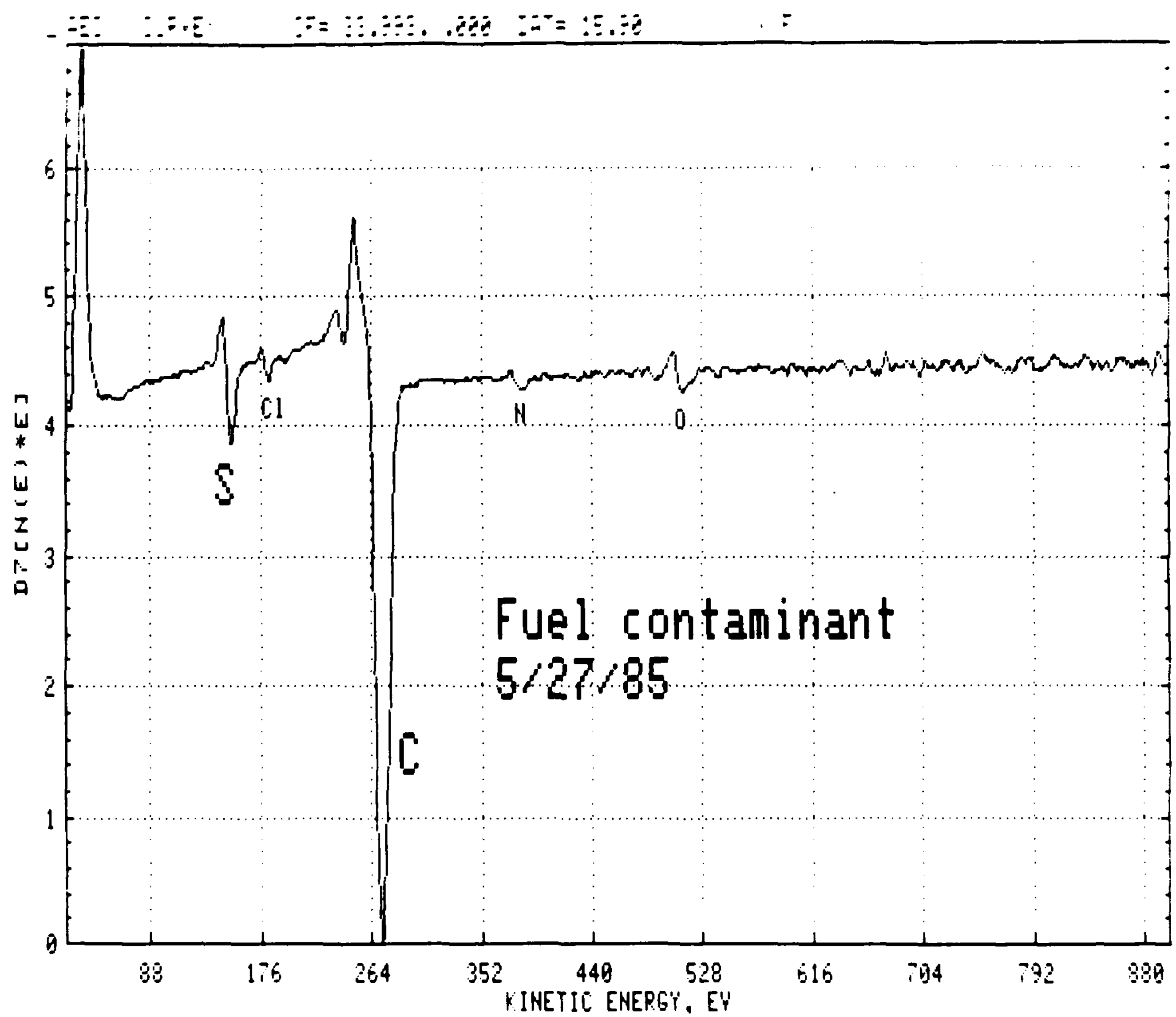


FIGURE 4. TYPICAL AUGER SPECTRUM OF A FLAKE RETRIEVED FROM SAMPLE AL-14205-T

Samples of flake material, sandwiched between KBr discs, were submitted for analysis using a microscope attachment in conjunction with Fourier Transform Infrared Spectrometry (FTIR). This apparatus allowed a spectrum to be obtained on as small as a 10 X 10 micrometer area. Several flakes were checked to ensure that a representative sample was used. The spectrum typical of these flakes is shown in Figure 5. A brief summary of chemical bond types represented by the stronger absorption bands is as follows: the broad band centered on 3500 cm^{-1} represents O-H bonds and is most likely due to moisture present in the KBr. Bands at 2957, 2925, and 2856 are C-H stretching vibrations of the aliphatic type CH_3 and CH_2 groups. The strong band at 1597 could be due to aromatic groups, amide II groups, or alternately could represent the COO^- group. The strong band at 1437 is most likely due to C-H deformations.

Laser Raman spectroscopy was also performed, but gave little additional information; only the presence of C-H and C=O groups could be established.

Additional samples were tested by Electron Spectroscopy for Chemical Analysis (ESCA). Results from ESCA analysis (tabulated in Table 2) of one flake gave 16 percent oxygen and 2.7 percent nitrogen, with the balance being carbon. Of the carbon, 55 percent was aliphatic or aromatic bonds, 32 percent C-O, 9.1 percent C=O, and 3.8 percent carboxylic acid or ester. The second flake analyzed was similar in elemental content, with 18 percent Oxygen, 0.9 percent Nitrogen, and 0.7 percent Iron, and the balance being carbon. These results are normalized to 100 percent for the elements observed; hydrogen is not detectable. Charging of the second particle prevented bond energy measurement. These data are consistent with phenol resin structures.

Analytical data do not contradict the visually inspired suggestion that the origin of the flaky material is some liner material which was in contact with the fuel. The presence of nitrogen is consistent with the range of nitrogen from amide and amine curing agents used in epoxy resins. The (presence of) C=O and C-O bands suggest an anhydride co-reactant curing agent or an amide. The aromatic component could have been derived from bisphenol-A which is reacted with epichlorohydrin to produce the resin prepolymer called the

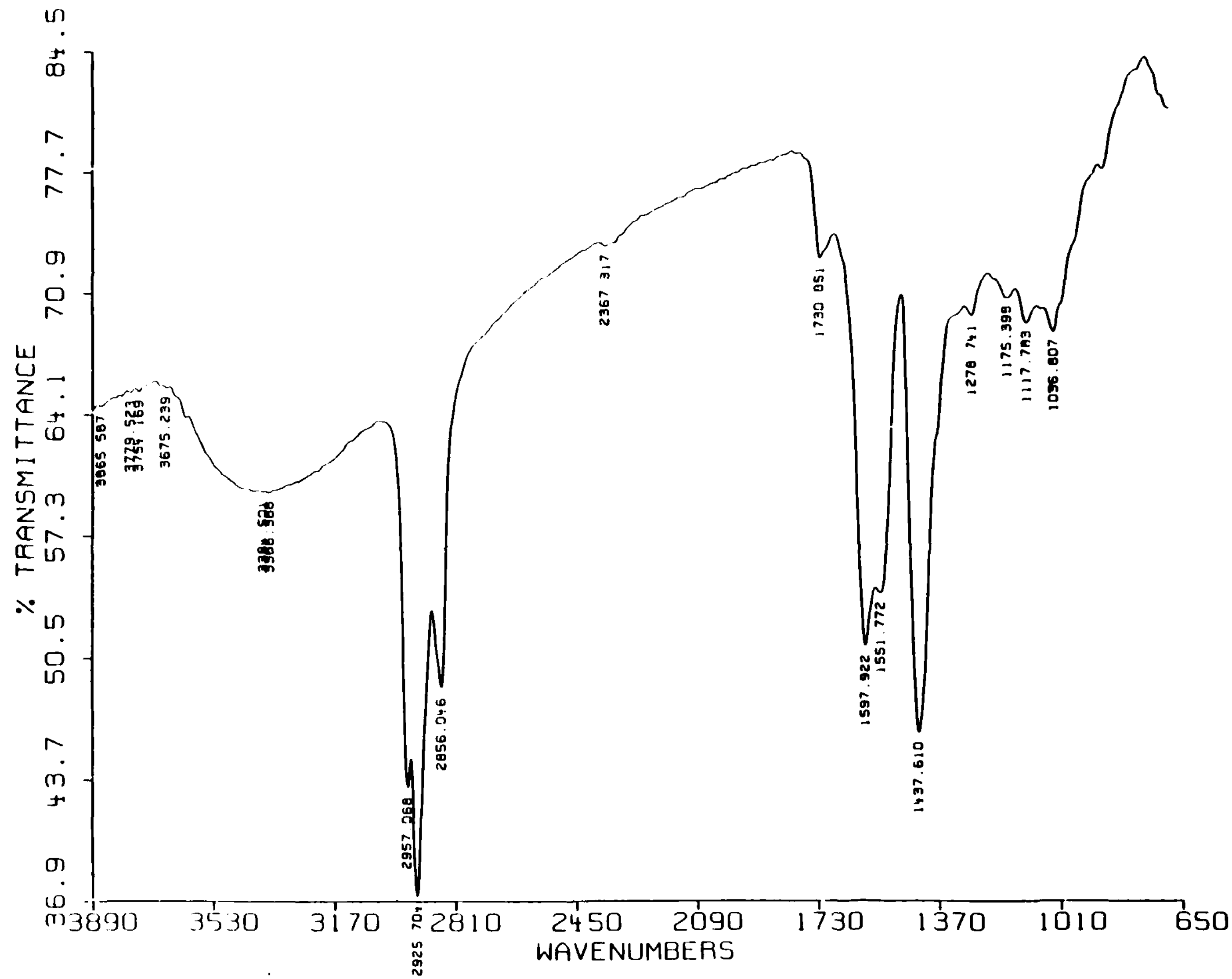


FIGURE 5. TYPICAL FTIR SPECTRUM OF A FLAKE RETRIEVED FROM SAMPLE AL-14205-T

TABLE 2. ESCA RESULTS FOR TWO FLAKES

Elements Detected	Flake Sample 1	Flake Sample 2
Oxygen	16%	18%
Nitrogen	27%	0.9%
Iron	0	0.7%
Carbon/Hydrogen	Balance	Balance

Carbon Bond Types	Flake Sample 1	Flake Sample 2
Aliphatic and Aromatic	55%	--
C-O	32%	--
C=O	9.1%	--
Carboxylic Acid	3.8%	--

"phenoxy resin." Vinyl esters are also a possible entity made by reacting epoxy resins with acrylic or methacrylic acid; however, the FTIR data indicate phenol-epoxy resin is more likely.

Samples of the flake material subjected to pyrolysis followed by gas chromatography/mass spectrometer analysis indicated large amounts of carbon dioxide (CO_2), m/e 44. A summary of the component species detected by mass spectrometry analysis is presented in Table 3. The total ion chromatogram and copies of mass spectra are available on request.

TABLE 3. TENTATIVE MASS SPECTROGRAPHIC IDENTIFICATION OF COMPONENTS DETECTED FROM PYROLYSIS OF "DANDRUFF FLAKES"

Scan No.	Tentative Identification
50-128	CO_2
413	hydrocarbon like
586	styrene
622	hydrocarbon like
709	benzaldehyde
755	phenol
765	hydrocarbon like
831	similar to alkane
859	hydrocarbon like coeluting with unidentified aromatic
874	2(3H)-furanone, Dihydro-3-methyl
916	methyl phenol
938	hydrocarbon like - possibly alcohol
945	unidentified
961	hydrocarbon like
1021	benzeneacetonitrile
1072	benzoic acid
1097	naphthalene with hydrocarbon like coeluter
1102	hydrocarbon like - possibly alcohol
1126	hydrocarbon like
1136	unknown
1162	unknown
1257	hydrocarbon like - possibly alcohol
1282	hydrocarbon like
1300	1,3-isobenzofurandione
1366	unknown
1403	hydrocarbon like - possibly alcohol
1513	chlorinated hydrocarbon
1517	hydrocarbon like - possibly alcohol
1639	1H-pyrazole, 3-methyl-5-phenyl - with coeluter
1540	hydrocarbon like - possibly alcohol
1633	unknown
1670	hydrocarbon like - possibly alcohol
1695	unknown aromatic
1699	hydrocarbon like

IV. RESULTS OF OTHER SAMPLES EXAMINED

The remaining fuel samples received were not given as detailed an evaluation as fuel AL-14205-T due to time and funding constraints. All fuel samples were examined visually per ASTM D 4176, and selected fuel samples were tested per Appendix A of MIL-T-5624L, for total solids and filtration time. Results of these tests are shown in Table 4. All fuels tested were within specification for total solids and filtration times. In all cases, the fuels exhibited poor visual appearance due to flake-like particles, lint, fibers, and other assorted debris. The fuel samples, helicopter filters, filter/separator cartridges, and other materials submitted for evaluation are discussed individually in the following subsections.

A. Fuel Sample AL-14091-T

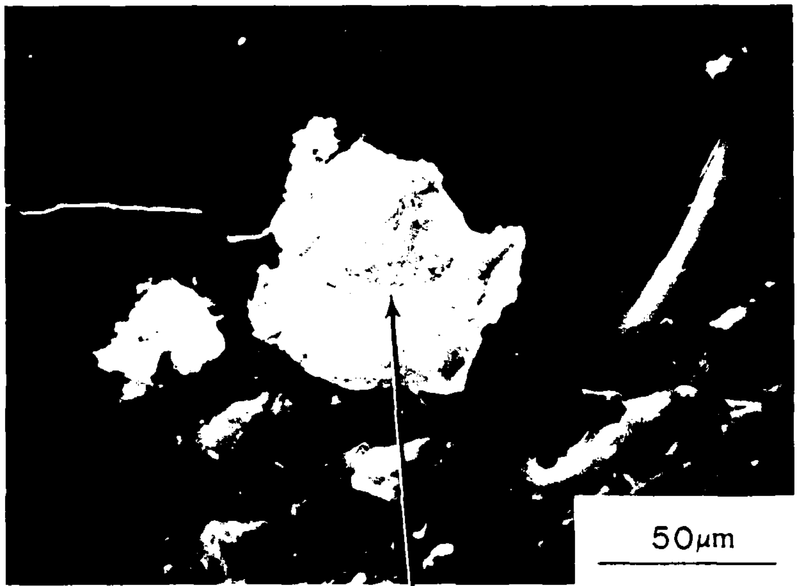
(Fort Knox Sample, From Tank Truck No. 15136 Bottom)

A clear, thoroughly-cleaned glass sample bottle was filled with approximately 750 mL of sample fuel and inspected in the lightbox. The presence of many flake-like particles was confirmed. Although some flakes had a similar appearance to the white-to-clear material noted in Sample AL-14205-T, most of the flakes appeared metallic in nature. After filtering to determine total solids, the millipore membrane filter was examined to determine types of materials present. Visual inspection of the filter at low magnification (10X to 40X) revealed the presence of many flake-like particles and assorted fibers. Most of the flake-like particles were metallic in color, and some of the approximately 20 fibers were brightly colored, as if dyed. Small particulates that appeared to be dirt and rust were also present.

A portion of the filter with representative contaminant materials was cut away and prepared for SEM/EDAX examination. Most of the flakes proved to be metallic, the majority of which were iron. An illustration of a typical iron flake and its associated X-ray spectrum are presented in Figure 6. Other metallic flakes proved to be lead/tin (Figure 7) and are possibly solder residue from the sampling can. One flake proved to be nearly pure nickel (Figure 8). The small pieces of dirt-like particulate were rich in Silicon,

TABLE 4. SUMMARY OF LABORATORY ANALYSIS

	ASTM D 4176	APPENDIX A, MIL-T-5624L		ASTM D 3114
Sample Identification	Visual	Total Solids, mg/L	Filtration Time, min	Fuel Electircal Conductivity, pS/m
AL-14090-T (Ft. Knox Tank Truck No.15136 - Middle Sample)	Many Flakes, Fibers & Particles Visible	0.5	--	340
AL-14091-T (Ft. Knox Tank Truck No. 15136 - Bottom Sample)	Many Flakes, Fibers & Particles Visible	0.3	4	345
AL-14171-T (Ft. Campbell Tank 202 - Lower 1/3)	Many Flakes, Fibers & Particles Visible	0.4	4	--
AL-14235-T (Ft. Knox Tank Truck No. 117 - Composite Sample From Compartment One)	Many Flakes, Fibers & Particles Visible	0.3	4	--
AL-14247-T (Ft. Knox Tank Truck No. 117 - Compartment One Sample Taken From Manifold	Many Flakes, Fibers & Particles Visible	0.5	4	--



IRON FLAKE

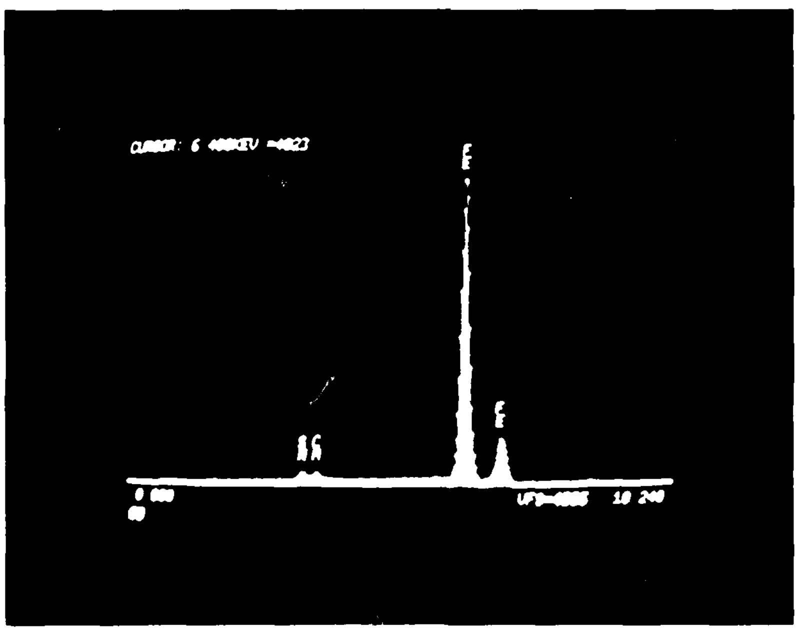
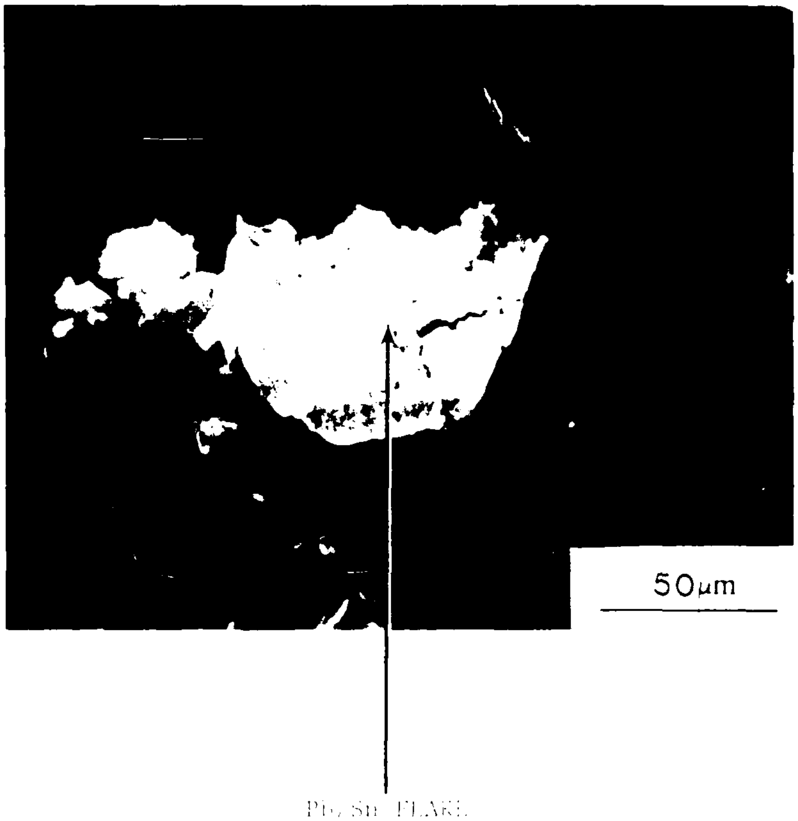


FIGURE 6. SEM PHOTOMICROGRAPH AND EDAX SPECTRUM OF TYPICAL IRON FLAKE RETRIEVED FROM SAMPLE AL-L-091-T



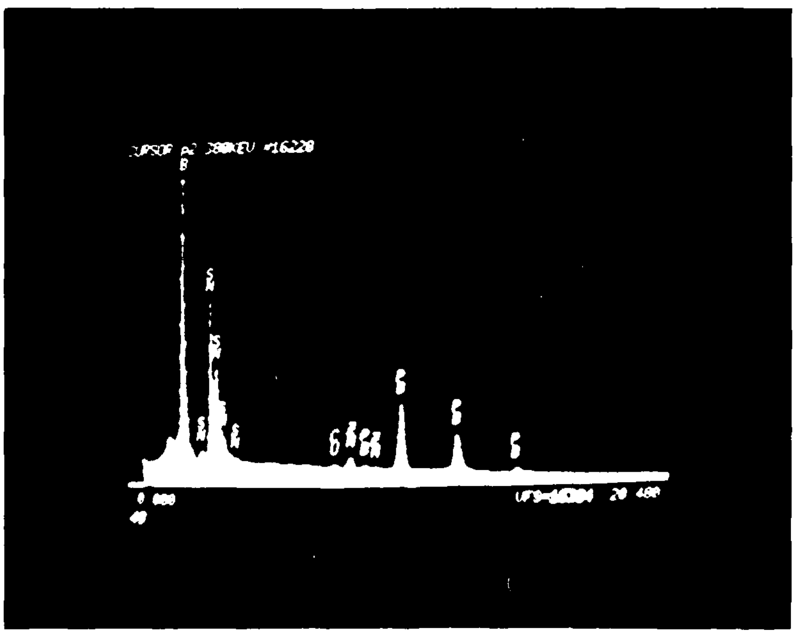
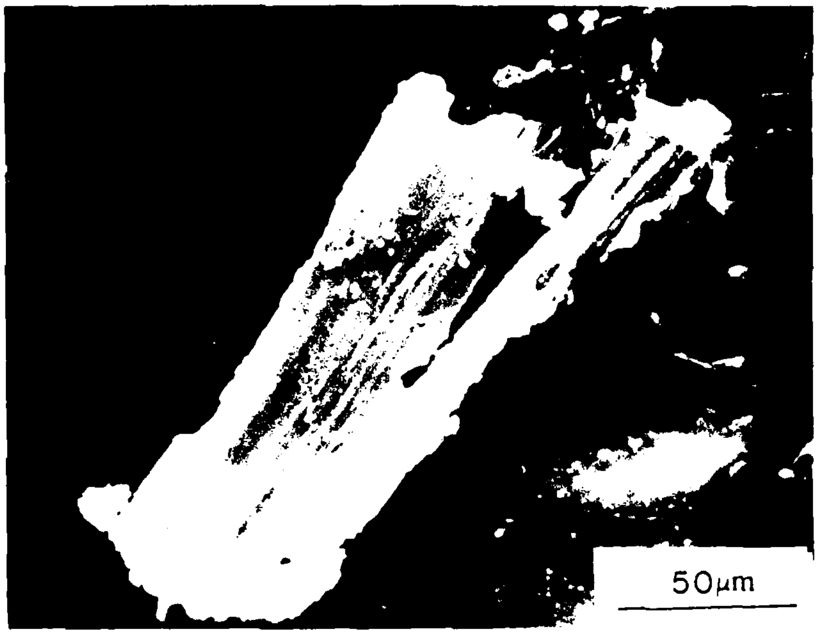
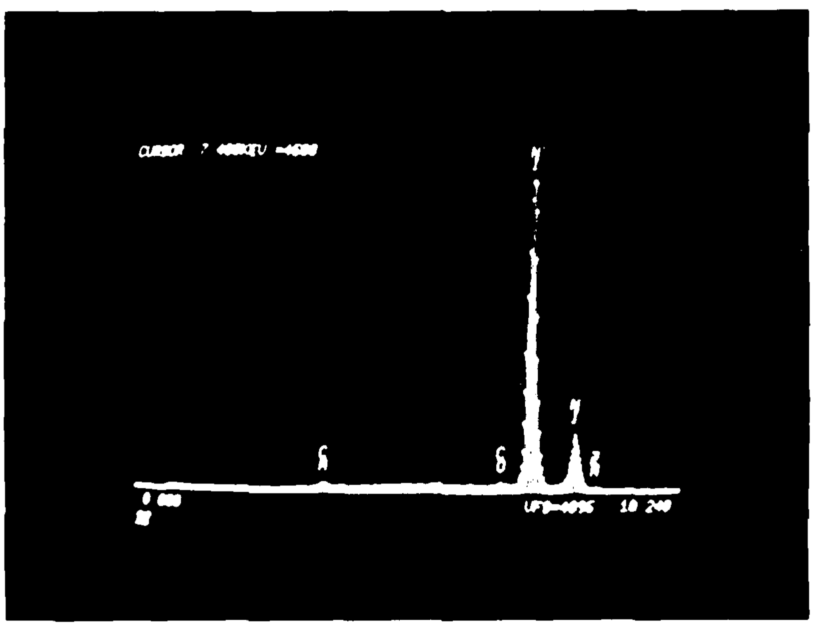


FIGURE 7. SEM [illegible] OF TYPICAL
[illegible] 31-14091-T





[illegible] PHOTOMICROGRAPH AND EDAX SPECTRUM OF NICKEL
[illegible] FROM SAMPLE AI-1-091-T

Calcium, and Aluminum, indicating that they were probably common clays. A very few flakes produced spectra with a high background in the low-energy range, indicative of organic material. These flakes appeared visually similar to the ones present in Sample AL-14205-T.

Most of the fibers were consistent with the appearance of natural fibers, such as cotton. A few of the fibers were round, smooth, and regular in shape (Figure 9). EDAX spectra of these round fibers showed high concentrations of Si, indicating fiberglass.

The inability to detect the many organic flakes on this filter is probably due to their being "buried" under the other debris, since flakes of this type were visually noted in the fuel sample prior to filtration.

B. Fuel Sample AL-14247-T
(Fort Knox Sample, From Tank Truck No. 117, Compartment 1, Manifold)

A portion of the fuel sample was visually inspected in the lightbox. Visual evaluation noted flakes, fibers, and dirt-like debris. One gallon of the fuel sample was filtered, and the resultant millipore filter was inspected under a stereo-microscope. Approximately 25 fibers of various lengths and colors were noted. These fibers all appeared to be cellulose-like material fibers, probably cotton, though a few of the smaller ones may be common lint or dust.

The entire millipore filter was prepared for SEM/EDAX evaluation. Flakes and debris examined showed only metals (Fe, Al, Cu) and clay-like particulate spectra. Flakes were noted during the visual inspection of the fuel which appeared similar to the organic "dandruff-like" flakes. However, they could not be detected on the membrane filter. It was possible that the filtration pressed these flakes tightly to the millipore surface to be covered by other debris. It was also possible that the concentration of debris on the filter surface contaminanted the flake so severely with inorganic material that it was impossible to resolve their organic nature.

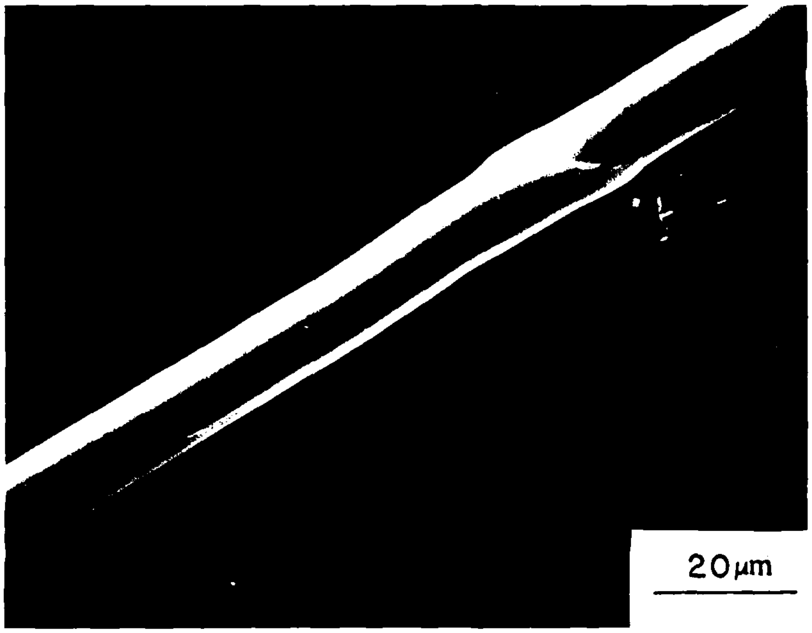


FIGURE 9. SEM PHOTOMICROGRAPH OF TYPICAL GLASS FIBER RETRIEVED FROM SAMPLE AL-14091-T

C. Fuel Sample AL-14171-T

(Fort Campbell Sample, From Tank No. 202, Lower One-Third)

A sample of the fuel was visually inspected in the lightbox. Several "flake-like"particles were evident along with some lint fibers and numerous smaller particles (<50 micrometers) of various colors and textures.

Approximately 1 gallon of the fuel was filtered through a millipore filter (Appendix A of MIL-T-5624L), and the filter was inspected under a stereo-microscope. Several "flake-like" particles were retrieved from the millipore with tweezers and mounted for SEM inspection and elemental analysis. Analyses of the individual particles showed high Ca in all cases and high S in one case. All particles also showed traces of Zn, Fe, Si, Al, and some contained traces of Cl, Ti, and Cu. It could not be readily determined if these were organic flakes coated with inorganic debris, or if the flakes were inorganic as the detected elements could indicate.

D. UH-1 Helicopter Filter

(UH-1 Fuel Filters From Fort Campbell--AL-14172-X)

Two UH-1 helicopter filters (pleated paper) were received from Fort Campbell. One filter was cut open to inspect the element. From this element, a number of particles were retrieved that exhibited similar phyical characteristics to the unusual "flakes." These particles were examined in the SEM, and chemical analysis was performed utilizing Energy Dispersive Spectrometry. Four of the samples showed a high background in the low-energy range, indicating the sample to possibly be organic. All these samples showed varying trace amounts of elements commonly found in fuel samples of this type (Si, S, Cl, Ca, Zn, Al, and Fe). Two samples were inserted into the auger spectrometer, but as soon as the beam was energized, the particles melted and volatilized. This indicates that the material present on the UH-1 filter is not the same as the "dandruff-like" flakes which were stable in the auger environment. The material which melted in the auger was more likely a waxlike material or thickener from assembly grease, etc. Thus, no evidence of the "dandruff flakes" was present in these fuel filters.

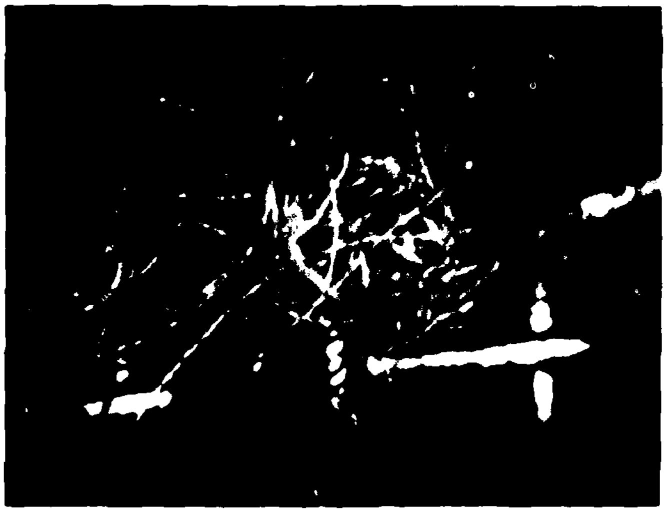
E. UH-60 Helicopter Filters
(From Fort Campbell)

Two used UH-60 helicopter fuel filters and a sample of "lint" from a third UH-60 fuel filter from the helicopter that flamed out, were submitted for BFLRF examination by Fort Campbell personnel. The lint was examined by SEM and had texture and shape characteristics of natural fibers (cotton). Samples of fibers from the exterior cover of two filter/coalescer elements representing different manufacturers were also submitted to determine if their fibers resembled those fibers found on the UH-60 fuel filters. The cotton fiber types found on the two sock-type filter covers were identical to each other and identical to the "lint-like" cotton fibers found on the UH-60 fuel filters. This does not infer, however, that they are the source of the fibers since cotton fibers from a variety of sources are a relatively common source of contamination. The UH-60 fuel filters were further examined for other contaminants in the depth grooves. Some small gel-like particles and a small loose tuft of fibers were found (see Figure 10). The fibers proved to be cotton when examined by SEM. The gel-like particles were probably a waxlike material or assembly grease. These particles could not be examined by SEM/EDAX because they volatilized in the vacuum system of the instrument under electron beam heating. No dandruff-like flake was found to be present on these fuel filters.

F. Filter Separator Element Sample AL-14173-X
(From Fort Campbell)

A filter separator element was selected for examination from a set of five received from Fort Campbell. The element was sectioned longitudinally and the interior surfaces examined at 10X to 40X magnification. Small flakes visually the same as the "dandruff-like" flakes found in fuel Sample AL-14205-T were detected in the filter element. The flakes were fragile, and attempts to remove them intact proved difficult. Flow tests of one of these elements at Kelly Air Force Base showed no restricted flow; however, this is a common occurrence when plugged elements are allowed to dry. Visual inspection of the filter separator elements did not reveal any deterioration or

A. GEL-LIKE PARTICLE



B. FIBER CLUMP

FIGURE 10. PHOTOMICROGRAPH OF GEL-LIKE PARTICLE AND FIBER CLUMP FOUND ON UH-60 HELICOPTER FILTER

manufacturing flaws which may have contributed to any problems. No further examination was made of the filter separator elements; however, they are being retained for future evaluation.

V. CONCLUSIONS

Many common types of debris were encountered during the course of this investigation. Most of these were materials commonly found in fuel samples (metal flakes, rust, fibers, dirt, etc.), but the flake-like organic debris was unusual to find in a fuel sample. The various analytical techniques indicate that this contaminant is probably a manmade (synthetic) organic component and is not a fuel product. It has a high melting point (>200°C) and is insoluble in most common solvents. The physical appearance of the flake (thin and flat while floating in the fluid) indicates that the material was originally formed or deposited in a thin layer prior to its introduction into the fuel. The most likely source of a material with these characteristics is an unpigmented paint or protective coating from one of the epoxy or similar resin families. Such a material could be introduced into the fuel by debonding or delamination of the contaminant from any handling, storage, or transportation equipment that contains such a coating. The elements, chemical bonds, and functional groups detected by the various analytical tests are consistent with this conclusion. Considerably larger amounts of the contaminant material in a relatively "clean" condition would be required to allow more definitive identification as a specific "chemical" compound.

The flakes are filterable, and the relatively small total volume and mass which they represent in the fuel should cause no major problem with filter plugging. Their presence is, however, objectionable and fuel will continue to fail ASTM D 4176 visual test unless the source is identified and eliminated.

VI. RECOMMENDATIONS

The source of the flake-like organic contaminant should be isolated by determining the point in the chain of distribution at which the flakes were first detected. This would require careful visual examination of the fuel during the stages of distribution. Personnel who perform this visual examination should be extremely familiar with the appearance of this specific contaminant material so that other debris types (metal flakes, fibers, etc.) are not incorrectly identified as being the organic flake material.

If more specific chemical compound identification would aid in source determination, larger volumes of material could be collected to allow more definitive analytical approaches. However, the recovery of large numbers of "clean" flakes from the fluid and the SEM/EDAX screening to ensure that each flake was, in fact, organic would be a tedious process involving many man-hours of effort before any sizable quantity could be collected.

Fuels presently containing this material should still be usable if filtered to remove the material. Removal should be confirmed by requiring the fuel to pass the visual clean and bright requirements. Of course, care should be taken not to introduce environmental contaminants during flushing of test containers or during sampling. Use of the light box for enhanced visual evaluation is highly recommended. It is also proposed that this enhenced "Clean and Bright" test procedure be considered by the U.S. Air Force for inclusion in the next revision of MIL-T-5624 fuel specification.

APPENDIX A

LETTER REPORTING FUEL CONTAMINATION PROBLEM

DEPARTMENT OF THE ARMY LEPERA/mr/AV354-3435

US ARMY BELVOIR RESEARCH & DEVELOPMENT CENTER
FORT BELVOIR, VIRGINIA 22060-5606

REPLY TO ATTENTION OF

16 APR 1985

STRBE-VF

SUBJECT: Fuel Contamination Problems at Smyrna, Tennessee

Commander
US Army General Materiel & Petroleum Activity
ATTN: STRGP-FT
New Cumberland Army Depot
New Cumberland, PA 17070

1. Reference:

a. MSG PT00262, USAGMPA, STRGP-FT, 211114 Mar 1985, subject: Suspected Fuel Filter Element Deterioration.

b. MSG PT 00180, USAGMPA, STRGP-FT, 081432 Apr 1985, subject: Contaminated Receipts of JP4.

2. In response to the reference messages which have identified a fuel contamination problem, this office was requested to provide technical assistance in defining the problem's origin and giving recommendations as to the necessary follow-up actions for disposal of the contaminated fuel supplies.

3. Selected fuel samples, fuel quality monitors, and filters were received from the Army Aviation Support Facility #1, TN ARNG, Smyrna, TN. These samples were taken in response to reported visual contamination of JP-4 Fuel (MIL-T-5624) from storage tanks and refueling vehicles. Operating personnel and Quality Assurance (QA) representative reported seeing fibers suspended in the fuel along with other particulate debris. Personnel from this Center were initially contacted to determine if the DoD Standard Filter/Separator (F/S) coalescer elements were the source of the fiber contaminant. The samples were subsequently taken on this presumption.

4. The samples which were forwarded to this Center consisted of Matched-weight Membrane Monitors, samples of JP-4 fuel, and F/S elements from the suspect refueler vehicle. There was however insufficient fuel sample for conducting a detailed analysis as to the origin of the reported fiber-like contaminant. Since that time however, a 5-gallon sample of the "contaminated" JP-4 fuel has been sent to the US Army Fuels and Lubricants Research Laboratory. Using this volume of sample, an identification as to the origin of the contaminant should be possible.

STRBE-VF
SUBJECT: Fuel Contamination Problems at Smyrna, Tennessee

5. The analyses performed on the samples forwarded to this Center have been completed. The results obtained on the Matched-weight Membrane Monitors are provided on Encl 1. The results obtained on the four samples of JP-4 are provided on Encl 2. The results on the F/S elements are given on Encl 3. Enclosures 4-6 provide color photographs (i.e., Figures 1 through 5) of the debris mentioned in the test results.

6. Specific recommendations were requested as to the contaminated JP-4. The fiber-contaminated fuel can probably be cleaned by the use of specialized decontamination equipment. The Military Standard filter coalescer elements are essentially mat filters designed to coalesce water and remove suspended solids of a nominal micron size. While most particulates are removed by these filters, it may be possible that certain types will not be removed. A micronic or screen filter of a given micron rating would remove all particles of that size and larger. Such filters are commercially available although they usually do not fit into military F/S vessels. Another alternative would involve multiple passes through the current F/S units. However, care should be taken to prevent build up of static electricity in the fuel. This Center will be willing to provide any technical assistance relative to reclaiming this fuel.

7. A more detailed letter report on testing with different F/S elements which has been completed by the Logistics Support Laboratory will be forthcoming shortly.

8. Point of Contact for further questions relative to the above is Mr. W. Williams, this office (AV 354-3576).

9. TROSCOM - PROVIDING LEADERS THE DECISIVE EDGE.

FOR THE COMMANDER:

Emil J. York

6 Encls
as

EMIL J. YORK
Director, Materials, Fuels and
Lubricants Laboratory

CF:
CDR, Defense Fuel Supply Center, ATTN: DFSC-TB (Mr. Carley), Cameron Station, Alexandria, VA 22314
CDR, US Air Force Wright Aeronautical Laboratory, ATTN: AFWAL/POSF (Mr. Churchill) Wright-Patterson Air Force Base, OH 45433
CDR, San Antonio Air Logistics Center, ATTN: SAALC/SFT (Mr. Makris), Kelly Air Force Base, TX 78241
US Army Fuels and Lubricants Research Laboratory, ATTN: Mr. Sid Lestz, Post Office Drawer 28510, San Antonio, TX 78284
CDR, US Army Natick Research and Development Center, ATTN: STRNA-YE (Mr. Rogers), Natick, MA 01760
LTC Robert D. Whitworth, US Army Aviation Support Facility #1, TN Army National Guard, Bldg 603, Smyrna Airport, Smyrna, TX 37167

MONITOR SAMPLE TEST RESULTS

Type of Sample. Millipore Matched-Weight Monitors utilizing 0.8μ membranes for gravimetric determination of particulates. Millipore Contamination Analysis Monitors utilizing 0.8μ membranes with 1/8" grid for counting of fibers. One liter of fuel had been passed through each monitor.

Sample Identification and Sample Site.

1. Sample "A" - Sampled from JP-4 refueler CD 6383 containing "contaminated" JP-4 (i.e., with fibers), after passage through F/S containing new Banner filter/coalescer elements.

2. Sample "B" - Sampled from same refueler and F/S but also containing Facet GO-NO-GO fuses.

3. Sample "C" - Sampled from same refueler after passage through F/S Containing new Keene filter/coalescer elements (from Fort Campbell, KY).

4. Sample "D" - Sampled from same refueler and F/S but also containing Keene GO-NO-GO fuses.

Tests Performed. Particulate matter per ASTM D 2276; "Fiber Count" based on procedure in MIL-F-8901, Appendix D.

Results.

"A" - No particulates indicated via gravimetric determination. Microscopic examination of Monitor membrane indicates no fibers or any significant debris.

"B" - No particulates indicated via gravimetric determination. Microscopic examination of Monitor membrane indicated one fiber (white; about 400 microns long) but no other significant debris.

"C" - No particulates indicated via gravimetric determination. Microscopic examination of Monitor membrane indicates no fibers or any significant debris.

"D" - No particulates indicated via gravimetric determination. Microscopic examination of Monitor membrane indicated six (6) fibers (white; see Figure 1) but no other significant debris.

Comments. Results indicate clean fuel and that the filter-coalescers were probably working properly.

FUEL SAMPLE TEST RESULTS

Type of Sample. One-gallon metal containers of JP-4 meeting MIL-T-5624.

Sample Identification and Sample Site.

1. Sample "1" - Sampled from JP-4 underground fiberglass tank using a Bacon Bomb sampler near bottom; fuel apparently had not passed through a filter/separater unit.

2. Sample "2" - Sampled from same tank and is a composite of all levels.

3. Sample "3" - Sampled from same tank by dipping after recirculating approximately 200 gallons of fuel.

4. Sample "4" - Sampled the same as Sample 2.

Tests Performed. The following tests were performed:

- Workmanship or "Clean and Bright" determination as stated in MIL-T-5624
- Fuel conductivity per ASTM D 2624 to determine level of static dissipator additive (SDA)
- Determination of Fuel System Icing Inhibitor (FSII) using the B-2 test kit
- Filtraion Time and Total Solids per MIL-T-5624, Appendix A
- "Fiber Count" per MIL-F-8901, Appendix D

Results and Comments.

Workmanship:

All samples were clear and bright exhibiting no visible water. Some rust-like sediment was found in the bottom. Many fibers could be seen in suspension appearing to be about ½ mm long. Sample 3 appeared to have slightly less fibers.

Fuel Conductivity:

Sample 1 - 130 C.U.
Sample 2 - 140 C.U.
Sample 3 - 110 C.U.
Sample 4 - 200 C.U.

The relatively low values indicate low concentrations of the SDA.

Fuels System Icing Inhibitor (FSII):

Sample 1 - 0.11 vol %
Sample 2 - 0.10 vol %
Sample 3 - 0.10 vol %
Sample 4 - 0.11 vol %

These reflect normal values found in JP-4.

Filtration Time:

Sample 1 - 1 min 34 sec for 2000 mL
Sample 2 & 4 - 6 min 38 sec for 4000 mL
Sample 3 - 1 min 47 sec for 200 mL

Samples 2 and 4 were combined as they were thought to be from the same source. This allowed for sufficient fuel to run at least one complete test. It would appear that all values obtained are within specification limits (i.e., a maximum of 15 minutes for one gallon).

Total Solids:

Sample 1 - 0.4 mg/L
Samples 2 & 4 - 1.3 mg/L
Sample 3 - 0.1 mg/L

All values are essentially below the maximum specification limit except for the combined Samples 2 & 4. The failing value for this sample could not be explained.

Fiber Count:

Sample 1, based on 1000 mL
Samples 2 & 4, based on 1000 mL
Sample 3, based on 850 mL

All of the membranes were cluttered with fibers. An actual count was impossible because it was difficult to distinguish individual fibers due to their overlapping. It is estimated that each grid (75 to a membrane) held a minimum of 5 fibers giving the following estimated values -

Sample 1 = 375 fibers per liter
Samples 2 & 4 = 375 fibers per liter
Sample 3 = 440 fibers per liter

Other debris were also present including rust particles.

Photomicrographs showing typical fibers and associated debris are shown in Figures 2 and 3.

FILTER ELEMENT SAMPLE TEST RESULTS

Types of Samples. Filter coalescer elements (meeting MIL-F-52308) from the refueler vehicle containing the contaminated JP-4. The filter elements were manufactured by Banner.

Tests and Comments. Each of the six (6) filters were visually examined for visual defects and contaminants. All filters appeared normal and clean, although wet with fuel. One filter was tested in a coalescence tank to check for end-cap leaks and water coalescing ability; no anomalies were found. Another filter was cut open and its particulate section (inner pleated paper core) examined microscopically for retained fibers; none were found. Samples of both types of fiberglass (coarse and fine) used in the coalescing section of the filter elements were examined and photographed (Figures 4 & 5). Comparison of the photograph with those of the contaminant fibers (Figures 2 & 3) would appear to rule out the filter elements as the source of the contaminant.

Fig. [illegible]. (40X)

200μ

Fig. [illegible]. (40X)

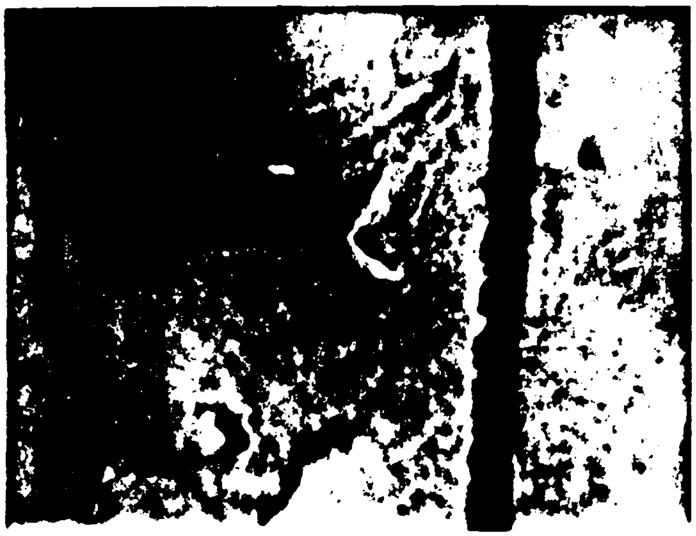


Fig. 3. (40X)

200μ

Fig. 4. (40X)

Fig. 5. (40X)

200 μ

APPENDIX B

ANALYSIS RESULTS OF JP-4 FUEL SAMPLES

U.S. ARMY FUELS AND LUBRICANTS RESEARCH LABORATORY

6220 CULEBRA ROAD – P.O. DRAWER 28510 PH:512-684-5111 SAN ANTONIO, TEXAS 78284

29 April 1985
File: 02-8341-199

USAFLRL

Commander
U.S. Army Belvoir Research and
Development Center
Attn: STRBE-VF, Mr. M.E. LePera
Fort Belvoir, Virginia 22060-5606

Subject: Laboratory Analysis of JP4 Fuel Samples from the Tennessee Army National Guard, Smyrna, Tennessee

Dear Sir:

1. AFLRL received five each one-gallon samples from the Tennessee Army National Guard at Smyrna, Tennessee, for analysis on 11 April 1985.

2. The fuel was analyzed for total solids and filtration time (MIL-T-5624, Appendix A), visual examination, cloud point, conductivity, and fuel system icing inhibitor. The results were as follows:

Total solids, mg/L	0.4
Filtration time, min.	4
Visual	White particles and fibers
Cloud point, °C	<-55
Conductivity, ps/m	150
Fuel system icing inhibitor, vol.%	0.09

3. The sample filter from the total solids analysis was further analyzed using a scanning electron microscope and an x-ray fluorescence spectrometer in an attempt to better identify the white particles present in the fuel. The results of these analyses are given in Attachment A.

4. If there are any questions, please contact Steven Westbrook at (512) 684-5111.

Very truly yours,

S. J. Lestz
Director

SJL/SRW/cgs (WD34.B)
Attachment
cf: LLS, SRW, JGB

ATTACHMENT A

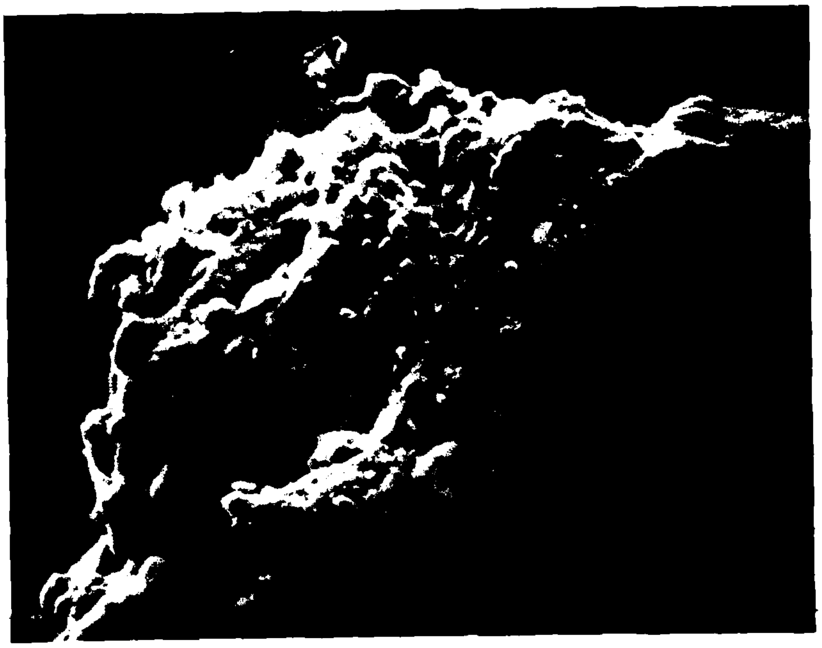
PARTICLE IDENTIFICATION

A number of contaminant particles were retrieved from fuel sample AL-14063T and visually categorized (40X stereo microscope) into five general types of contamination.

The optical characteristics of these particles are:

Type 1	Small black particles (soft)
Type 2	Small black particles (hard)
Type 3	Small, milky white flakes
Type 4	Clumps of lint-like fibers, adhered to a yellowish, waxy appearing substance
Type 5	Small, metallic looking flakes

The contamination was then further characterized by visual inspection in a scanning electron microscope (See Figures 1-5, for the 5 types, respectively), and elemental analysis by energy dispersive spectrometry (See Figures 1A-5A).



500X 50μM

Figure 1. High chlorine, probably in association with some lighter element

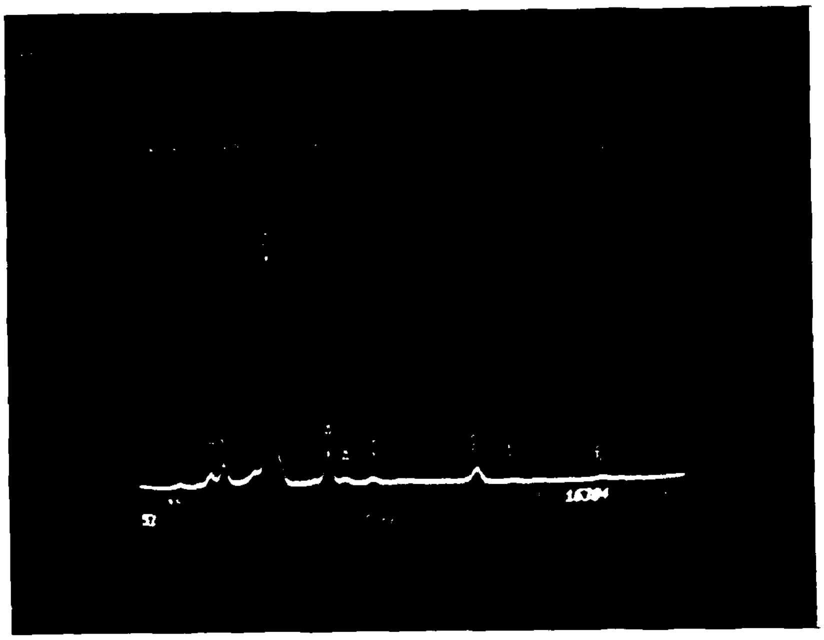


Figure 1A.

500X 50μM

Figure 2. Particle sample is comprised predominantly of lead, with appreciable amounts of other metals, including tin. This indicates that the particle could be a solder fragment, possibly from the sample can.

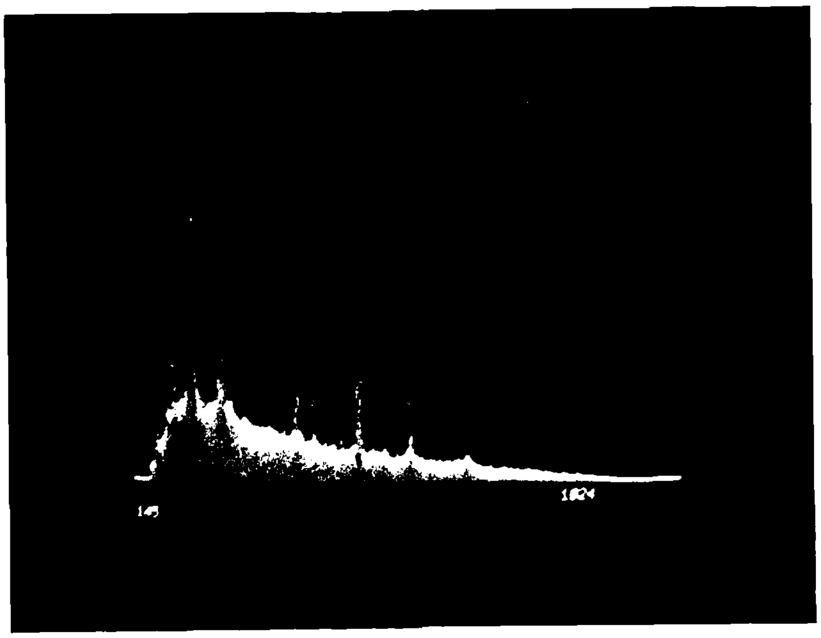


Figure 2A.

500X 50µM

Figure 3. High background in the low energy range indicates that this particle is composed mostly of organic molecules (possibly hydrocarbons). Physical appearance suggests that it is possible a man-made. non-woven fabric fragment.

Figure 3A.

300X 50μM

Figure 4. High background in the low energy range indicates that this sample is mostly organic. Physical appearance suggests lint or paper fibers.

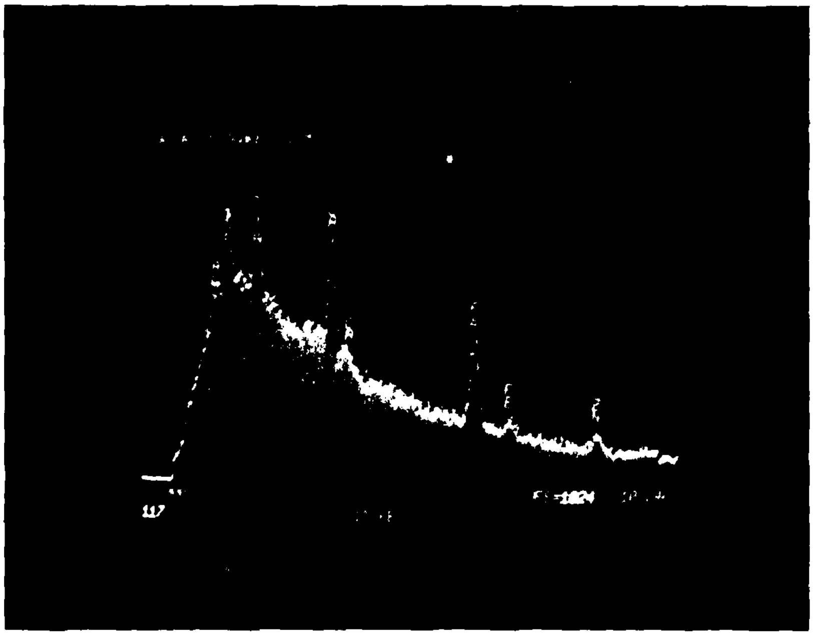


Figure 4B.

500X 50UM

Figure 5. Relative pure (low alloy) aluminum.

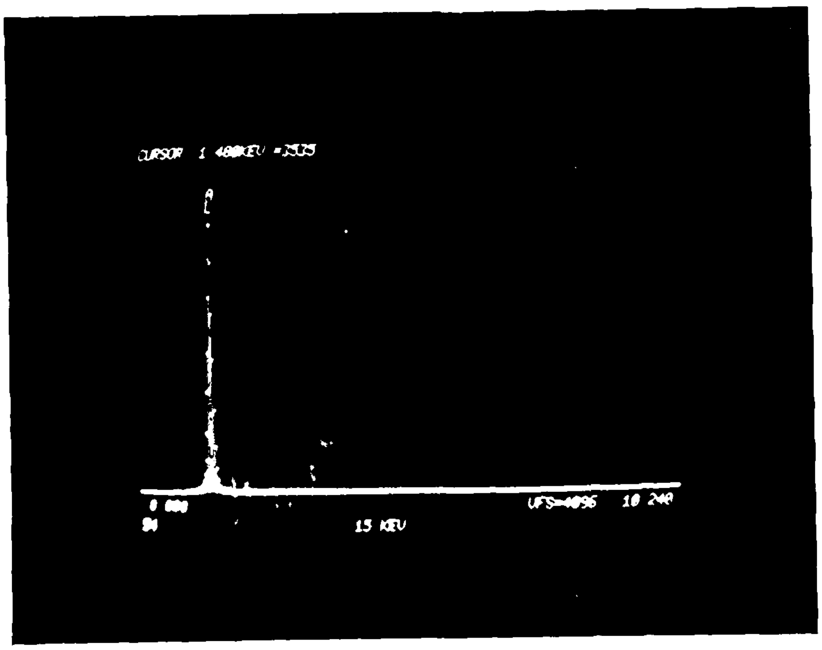


Figure 5B.

APPENDIX C

MEMORANDUM CONCERNING JP-4 FUEL CONTAMINATION

RECEIVED
APR 0 9 1985
AFLRL

STRBE-VF 3 April 1985

MEMORANDUM FOR RECORD

SUBJECT: JP4 Fuel Contamination Problem and User Assistance by BRDC

1. Reference:

a. MSG PT 00262 GMPA, STRGP-FT, 211114Z Mar 85, subject: Suspected Fuel Filter Element Deterioration.

b. MSG PT 00419 GMPA, STRGP-FT, 292030Z Mar 85, subject: Off Specification Receipts of JP4 at Fort Campbell.

c. Memorandum for Commander, BRDC, 27 Mar 85, subject: Response to User.

2. Reference 1a identified a problem causing a flame-out to occur on a UH-60 operating at Fort Campbell. The fuel filter was reported to contain excess fibers which were to have originated from a filter separator element. Reference 1b provided a follow-up and noted that Fort Campbell has since refused some fourteen tank truck shipments of JP4. [redacted] The rejection of these shipments was because of their failing the workmanship clause; i.e., the product contained "white suspended matter and excess fibers". Reference 1c provided a brief Memorandum surfacing fuel contamination problems occurring at a Tennessee National Guard facility located at Smyrna, Tennessee.

3. As was noted in Reference 1c, this Center was contacted by GMPA on 22 Mar 85 to provide assistance in resolving this problem.

4. Since the initial contact with this Center, a considerable amount of effort has been expended by personnel from both the Logistics Support Laboratory and the Materials Fuels and Lubricants Laboratory in attempting to identify the source of the problem. To date, the following facts are given:

a. The fuel in question being provided to both Fort Campbell and the Smyrna National Guard facility was refined by [redacted]. A DLA Quality Assurance Representative has witnessed the testing of the [redacted] JP4 and found the product to be acceptable for shipment.

b. The "contaminant" appears to be something that is being introduced in transit.

c. Samples of the suspect fuel provided to this Center are now being analyzed. All gravimetric tests show no debris and/or contamination. However, a visual swirling examination does indicate that minute particles may be present. Additional testing is being conducted in an attempt to isolate these minute particles.

STRBE-VF 3 April 1985

SUBJECT: JP4 Fuel Contamination Problem and User Assistance by BRDC

d. As per our recommendation, GMPA has requested the Smyrna National Guard personnel to ship samples of the suspect fuel to Natick R&D Center and the US Army Fuels and Lubricants Research Laboratory. The Natick personnel (i.e., Dr. Kaplan/STRNA-YE) will assess the fuel for microbiological organisms. The US Army Fuels and Lubricants Research Laboratory will utilize sophisticated analytical techniques in an attempt to isolate and identify the origin of these particles.

e. [redacted] has refused to ship any additional products until we are able to identify the source of this contamination.

f. Personnel at the Air Force Wright Aeronautical Laboratory have been contacted to solicit their assistance.

g. Based upon the work completed to date, it does not appear that the anti static additive (i.e., DuPont's Stadis 450) is the cause of the problem.

5. Personnel within this Center are continuing to work on this problem and will be in close communications with DLA and GMPA personnel. At this point in time, it is very difficult to point to the source of this problem.

Maurice E. LePera

Fuels & Lubricants Division
Materials Fuels & Lubricants Laboratory
Belvoir R&D Center

DEPARTMENT OF THE ARMY
US ARMY BELVOIR RESEARCH & DEVELOPMENT CENTER
FORT BELVOIR, VIRGINIA 22060

STRBE-GEQ

SUBJECT: Trip Report, 21-22 March 1985, Army Aviation Support Facility #1, TN ARNG, Smyrna, TN 37167

THRU: Chief, Fuel and Water Quality Branch

Chief, Engineering Division

TO: Director, Logistics Support Laboratory

1. Purpose: To investigate a report by TN ARNG personnel of JP-4 Contamination in their underground fiberglass storage tanks and their aircraft refueling tankers.

2. Personnel contacted:

LTC Robert D. Whitworth, Commander
Army Aviation Support Facility #1
Tennessee Army National Guard
Building 603, Smyrna Airport
Smyrna, Tennessee 37167
AV 694-3450 ext. 212
Comm (615) 252-3450

1 SGT William Prater
Army Aviation Support Facility #1
AV 694-3450 ext. 233,245

SSGT James L. Felts, Laboratory Technician
Tennessee Air National Guard
Fuels Management Office, FP6421
P.O. Box 17267
Nashville, TN 37217-0267

Mr. Osbia Jones, DAC
US Army, GMPA
New Cumberland, PA 17110
AV 977-6053/6445

STRBE-GEQ
SUBJECT: Trip Report, 21-22 March 1985, Army Aviation Support Facility #1, TN ARNG, Smyrna, TN 37167

3. Discussion: Mr. Jones and I were assigned by our respective offices to conduct this investigation. We began by checking refueler CD 6383 to make sure the filter elements and GO-NO-GO fuses were installed properly. After this was done, we took bottle samples of JP-4, after the filter-separator and visually examined them. Fibers were present in the fuel along with extremely fine (almost microscopic) debris. The filter elements and fuses were removed and new Banner elements (lot #029, DLA 700-81-C-0677) were installed. The fuses were not installed at this time. The JP-4 was then recirculated through the filter-separator and millipore fiber samples monitor "A" was taken along with a bottle sample. The fuel was clear and bright (no water) but it did contain numerous fibers and the same type of foreign matter seen earlier. Also, a few small drops of a clear liquid was observed floating on top of the fuel sample. Facet GO-NO-GO fuses were then installed using the same filter elements. The fuel was recirculated through the filter-separator and fiber sample "B" was taken along with a bottle sample (fiber samples will be analyzed at Ft. Belvoir, VA). The bottle sample was taken to the Tennessee Air Guard Laboratory in Nashville. It was visually examined and found to contain in excess of 10 fibers. The foreign matter was also visible in the sample.

Air Guard personnel stated they were not having any fuel contamination problems at all. They also stated that they obtained their JP-4 from a different supplier than the Army Guard at Smyrna. I asked to see their supply of filter elements in order to compare it with those used in Smyrna. Following is a list of the Air Guards elements followed by a list of the Army Guards elements that were used in its tankers.

AIR GUARD

BANNER	KEENE
DSA700-74-C-4180	DLA700-79-C-0516
DLA700-80-C-1730	DLA700-79-C-2835
DLA700-81-C-3984	DLA700-79-C-2836

ARMY GUARD

BANNER	KEENE
DSA700-74-C-4180	DLA700-82-C-3300
DLA700-79-C-0782	
DLA700-80-C-1730	
DLA700-81-C-0677	
DLA700-81-C-3984	
DLA700-84-C-1190	

STRBE-GEQ
SUBJECT: Trip Report, 21-22 March 1985, Army Aviation Support Facility #1, TN ARNG, Smyrna, TN 37167

The above test procedure was repeated in refueler CD 6383 with Keene elements (from Ft. Campbell) (2 ea lot #2962, 1 ea lot #3002, DLA700-82-C-3300) and Keene GO-NO-GO fuses. Fiber sample "C" was taken with elements only installed and fiber sample "D" was taken with elements and fuses installed. The JP-4 was examined visually and found to contain numerous fibers and the same type of foreign material observed in earlier samples. I then traced the procedure for handling JP-4 by the Army Guard at Smyrna. The fuel is delivered in commercial tankers and gravity off-loaded (no filter separator used) into old underground steel tanks formerly used by a commercial airline. From here, the fuel is pumped into Army National Guard tankers (no filter-separator used). The fuel is then gravity off-loaded (no filter-separator used) into the Guards three underground fiberglass storage tanks. From here it is pumped through a commercial filter-separator into a tanker. The tanker then delivers it through a filter-separator (with fuses) to the using aircraft.

Bottle samples of JP-4 were taken from the three fiberglass tanks. There was no method to recirculate the fuel in the tanks and obtain a representative sample of the fuel. The sample did contain some foreign matter and fibers but much less than I had anticipated. A quantitative value cannot be assigned to the samples. Fuel samples from these tanks are being furnished to Ft. Belvoir and to Air Force laboratories for complete analysis.

4. <u>Conclusions</u>: The investigation is continuing as to the cause/solution to the JP-4 contamination at Smyrna. Filter element tests will be conducted by this office. After these tests are completed and the JP-4 fuel samples are analyzed, corrective action will be recommended.

Ralph J. Polk

RALPH J. POLK, JR.
Mechanical Engineering Technician
Engineering Division
Logistics Support Laboratory

APPENDIX D

TRIP REPORT TO FORT CAMPBELL, KY

U.S. ARMY FUELS AND LUBRICANTS RESEARCH LABORATORY

6220 CULEBRA ROAD—P.O. DRAWER 28510 PH:512-684-5111 SAN ANTONIO, TEXAS 78284

File: 02-8341-199
28 May 1985

USAFLRL

Commander
U.S. Army Belvoir Research
and Development Center
Attn: STRBE-VF, Mr. M.E. LePera
Ft. Belvoir, Virginia 22060-5606

Subject: Trip Report for the Period 1-7 May 1985 to Fort Campbell Army Air Field, technical liaison visit regarding quality of fuel used in aircraft.

Dear Sir:

Attached trip report by L.L. Stavinoha for the period 1-7 May 1985 is forwarded for your information.

Please call if there are any questions.

Very truly yours,

Leo L. Stavinoha
for S. J. Lestz
Director

SJL/sjd
Attachment

TRIP REPORT

Leo L. Stavinoha
Fort Campbell, Kentucky
1-7 May 1985

Purpose: Technical Liaison visit to Fort Campbell Army Air Field regarding quality of fuel used in aircraft.

Persons Contacted:

Mr. Moody	Air Field Fuel Management
Joe Marable	Air Field Fuel Management
Mr. Yarboro	Fuel/Oil Laboratory
David Hansard	Fuel/Oil Laboratory
Col. Otis	Army Aircraft Maintenance 5th Trans. BN
Bill Tucker	General Electric Co. Technical Representative
Jerry Carter	Sarkarsky Technical Representative

Discussion:

Figure 1 provides a diagrammatic overview of jet fuel handling at Fort Campbell Army Air Field. Jet Fuel (JP-4, Mil-T-5624) is received into the air field POL (aboveground) storage tanks (3 each) by air field personnel under the control of Mr. Joe Marable. The air field is capable of dispensing fuel through filter separators at the POL tank farm (from Tank No. 308) and from two (flightline) pump stations with underground fuel storage tanks (fed from the POL storage tanks). The air field personnel generally fuel transient aircraft and Army tank wagons (no filter separators), which supply the Army's fuel storage tank located at a hot refueling station at the airstrip called the Oasis Pump Station. Army aircraft (helicopters) and tank and pump units are fueled at the Oasis fueling station. Tank and pump units (equipped with filter separators) are used for cold fueling of aircraft.

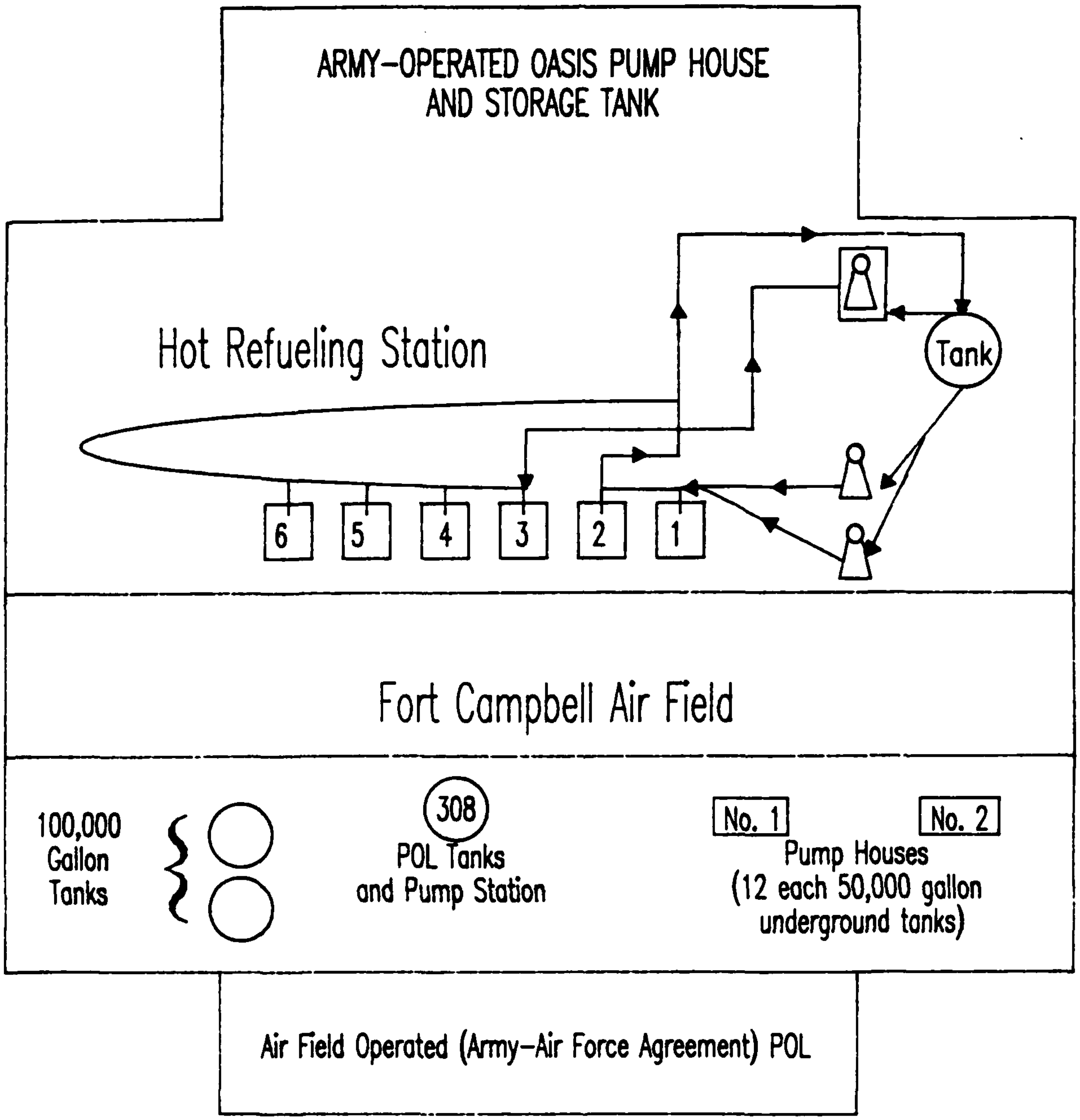


FIGURE 1. OVERVIEW OF JET FUEL HANDLING
AT FT. CAMPBELL ARMY AIR FIELD

Mr. Marable explained that fuel previously being delivered from an oil company failed the clean and bright workmanship requirement so badly (even though it was well within the filterable solids limits (4 mg/gal. max) and the filtration time limit (of 10 minutes max)), that suspect fuel was no longer being accepted. Both Mr. Marable and Mr. Hansard were concerned about the fuel for two reasons:

1. Both MIL-T-5624L and MIL-HDBK-200F require tank truck/tank car delivery fuel to be visually free from undissolved water, sediment, or suspended matter and shall be clean and bright, while the Air Force T.O. 42B-1-1 visual test states "no detectable contamination, i.e., excessive water, solids, or discoloring."
2. The "dandruff-like flakes" visible in the tank car fuel by the "clean and bright test" were also detected in aircraft fuel tanks, samples from pump house filter/separator units, and samples from tank/pump unit filter separators.

While normal lint (fiber) and "more dense" particles (such as sand, rust, etc.) were also present in the fuel, real concern was for the less dense "dandruff-like flakes" which were said to look like "flakes of candle wax" or "Teflon flakes" under a stereomicroscope.

A verbal, Wright Patterson Air Force Base, report on the analysis of filterable material from the suspect fuel (composed of samples from six tank trucks) indicated that it contained principally "trash or garbage," but that an attempt would be made to identify the "flakes" present in two 1-gallon samples from the Fort Campbell POL tankage.

Col. Otis provided a small sample of lint taken from the outer surface of a UH-60 helicopter (which had experienced a flameout) fuel filter and two UH-60 fuel filters retained during the recent replacement of all (133) UH-60 helicopter fuel filters. The material analysis report prepared by United Technologies Research Center (Attachment No. 1) indicated that fibers on three helicopter fuel filters were primarily cotton (cellulose). Additionally, two UH-1 helicopter fuel filters were provided to Mr. Yarboro for shipment to AFLRL.

Mr. Marable indicated that the Air Force F-16 aircraft, which crashed about 5 minutes after refueling at Fort Campbell, was fueled from Tank No. 202. Two other aircraft which fueled from Tank No. 202 that same day did not experience any problems. The filter separator elements from Tank No. 202 were removed since they showed a pressure differential of 19.5 lb (while 20 lb is the maximum allowable before replacement). Five 1-gallon samples were taken from the bottom third of Tank No. 202 (using a Bacon bomb sampler) and shipped to AFLRL. The five filter elements taken from the filter-separator (and a new element) were also shipped to AFLRL. Visual inspection of the filter elements did not show any unusual ruptures or distress of any of the filters.

The air field has R-5 tank and pump units for remote refueling. The filter separator units on the R-5's have 30 filter elements (4330-00-983-0998, DLA-700-80-C-1730). Three successive visual tests were made on one of the R-5 refuelers (No. 579). The sampler was thoroughly rinsed (approximately 1 quart), and each successive bottle (rinsed with approximately 1/3 quart fuel). No particles were observed in the first bottle, while one each fiber (particle) was observed in each of the two successive test bottles.

The pipe from Tank No. 308 was sampled during fueling of an Army tank wagon from filter separator No. 5. The gravimetric particulate was a 0.3 mg/L and a standard visual inspection showed some fibers and a few light flakes. Also, filter separator No. 5 was sampled with fuel from Tank No. 308 (during fueling of the Army tank wagon), giving a millipore monitor gravimetric value of <0.05 mg/L and a visual test of some fibers and one light flake. Two days later, the filter separator unit No. 5 was again checked by visual analysis. The millipore monitor sampler was thoroughly rinsed (with test fuel) as was each of three successive visual test bottles. The first bottle had 5 to 6 visible particles (small, possibly flakes), while each of the two remaining bottles had no visible particles. Using a Bacon Bomb sampler, 0.5 gallon and 1 gallon of tank bottom fuel sample were obtained in two standard cans. From the first 0.5-gallon sample filtered on a D 2276 pad, several of the waxy flakes were observed prior to drying the filter pad at 90°C for 22 minutes. After drying, the flakes appeared smaller, but did not melt. The second (1 gal.) sample was filtered by D 2276 and both of the millipore filters from the Tank 308 bottom fuel samples retained for analysis at AFLRL. Arrangements were made for two additional 1-gallon samples to be taken from the bottom of Tank No. 308 for shipment to AFLRL.

While fuel sampling and analysis of JP-4 at the air field are done by civilian employees, the fuel sampling and analysis at the Army's Oasis POL Station are done by U.S. Army personnel. Analyses are done in the 426th Supply and Service BN Petroleum Laboratory operated by Alpha Company, Class III Platoon. The physical laboratory is the same as that used by the civilian personnel. Fuel sampling is done primarily by the 561 BN of the 102 Quartermaster. All Army personnel contacted during this visit were found to be highly cooperative and dedicated to doing a good job. Any negative statements made in the following report should reflect on the quality of training provided rather than on the conscientious job performance of any individual soldier.

While clean bottles are furnished for sampling tank and pump units (required after tank filling before dispensing to aircraft), no provision was made for flushing the fuel hose nozzle (mounted with exposure to road dust on tank) during sampling. The first bottle from one tank and pump unit badly failed visually the first time with many small "flakes" present. The unit was recirculated for 1 hour, and a second sample was taken after flushing the nozzle into the tank fill hole (under a shed due to rain) into a JP-4 flushed sample bottle. Visual testing showed no particles or flakes.

General practice is to transport sufficient fuel on Friday evening to the Army Oasis POL Station (aboveground) tank to essentially fill the tank. On Saturday morning, the Oasis system is operated for 2 hours to recirculate the fuel and then sampled for analysis before fueling any aircraft. Sample volumes are 1 quart for visual analysis followed by FSII and conductivity analysis. After this sampling, a millipore monitor is installed in the sampler and 1 quart of fuel drawn through the filter monitor. At my request, on this occasion, 1 gallon of fuel was drawn through the monitor at each of three points; (1) Oasis tank bulk fuel, (2) pumphouse filter separator, (3) portable filter/separator No. 2 feeding hot fueling pads Nos. 1 and 2. On Friday morning, the following results were obtained:

Location	D 2276, mg/Gal	Visual
Oasis Tank	0.2	Lint, some flakes
Oasis Pumphouse Filter Separator	0.02	Dirty, >10 flakes
Portable Filter Separator No. 2	0.08	Clean
Portable Filter Separator No. 3	(Pump motor out of order)	

On Saturday morning after recirculating the practically full tank, I observed the sampling and the following results were obtained:

Location	D 2276, mg/Gal	Visual
Oasis Pumphouse Filter Separator	0.11	Dirty, many small flakes
Portable Filter Separator No. 2	0.10	Clean

I took a clean visual test bottle to the Oasis and with flushing obtained a clean visual test from the pumphouse filter separator. Ordinary dust, dirt, and human-handling debris "may" be showing up in the visual test due to handling and environment. The millipore monitor sampler at the Oasis does not have a grounded sampling tube and was not rinsed (nor was the bottle rinsed) with fuel prior to sampling during the first sampling Saturday morning. Greater precautions are required for safety and flushing of sampling equipment. Interpretation of MIL-T-5624 particulate by D 2276 (to be reported as mg/liter) has led to the use of 1-liter sample filtering rather than the 1-gallon minimum required by ASTM D 2276; clarification of the intent of MIL-T-5624 is required. Air Force practice seems to be to require 1 gallon sampling and reporting either as mg/gallon or mg/liter.

Conclusions:

1. Presence of unusual "dandruff-like flakes" in fuel at Fort Campbell Army Air Field was confirmed, at least in Tank No. 308, which received most of the "worst-appearing" fuel.

2. No strong evidence was found to substantiate suspicion that "flakes" were passing through filter separators even though flakes were present in fuel at the Oasis POL Station and in samples from helicopters.
3. Ordinary trash and system debris as well as unusual "wax flakes" were observed on test filters from the POL tank fuel. Emphasis should be placed on identifying the "wax flakes" rather than the lint, glass fiber, and inorganic debris.
4. The fragile nature of the "wax flakes" could lead to the flakes breaking apart in the filter separators and subsequently reforming through agglomeration in the filtered fuel at a later time (a highly hypothetical suggestion).

UNITED TECHNOLOGIES SIKORSKY AIRCRAFT

North Main Street
Stratford, Connecticut 06601
(203) 386-4000

April 16, 1985

Commander
5th Transportation Battalion
ATTN: AMO (CW4 H. Swain)
Fort Campbell, Kentucky 42223-5000

(Ltc O7's)

Mr. Swain:

I am forwarding the results of the analysis conducted by our Research Center on the three fuel filters returned to Sikorsky Aircraft on the 14th of March, 1985.

Please call me if you require any further assistance or information concerning this matter. My telephone number is Area Code (203) 386-3229.

Very truly yours,

UNITED TECHNOLOGIES CORPORATION

Wesley M. Shafer
Aircraft Safety Investigator
SIKORSKY AIRCRAFT DIVISION

WMS:nmp
Enclosure

United Technologies
Research Center

1935 /...

Material Analysis Report

Requestor: OAKES T   Date submitted: 3/18/85

MML No. : 05360

Description:

FUEL FILTER CONTAMINATION

Analysis:

IDENTIFY FOREIGN MATERIAL ON FUEL FILTERS

Results:

FIBERS FOUND ON THREE HELICOPTER FUEL FILTERS (S/N 23851 #1,23910 #1, 23910 #2) HAVE BEEN IDENTIFIED AS COTTON (CELLULOSE) BY SEM AND FTIR. TYPICAL SEM PHOTOGRAPHS AND FTIR SCANS ARE ATTACHED.

ASHING THE FIBERS REVEALED THAT THE MATERIAL WAS 77-95% ORGANIC, THE AMOUNT OF INORGANIC DEBRIS TRAPPED IN THE FIBERS ACCOUNTING FOR THE APPARENT RANGE IN ORGANIC CONTENT. AN EMISSION SPECTROGRAPH OF THE RESIDUE SHOWED THE COMPOSITION TYPICAL OF GRIT FOUND ON FUEL FILTERS: % FE=5, SI=>10, AL=10, MG=5, TI=5, CU=5, SN=1, ZN=5, CA=5, AND NA=2.

ANALYSIS OF A FOURTH FILTER, TAKEN FROM A FUEL TRUCK, FOUND THAT THIS FILTER WAS APPROX. 95% FIBERGLASS, WITH A SECTION OF WOVEN MATERIAL WHICH LOOKED LIKE COTTON. FTIR ANALYSIS OF THE WOVEN MATERIAL CONFIRMED THAT IT HAD A STRUCTURE SIMILAR TO THE COTTON FIBERS FOUND ON THE ENGINE FILTERS.

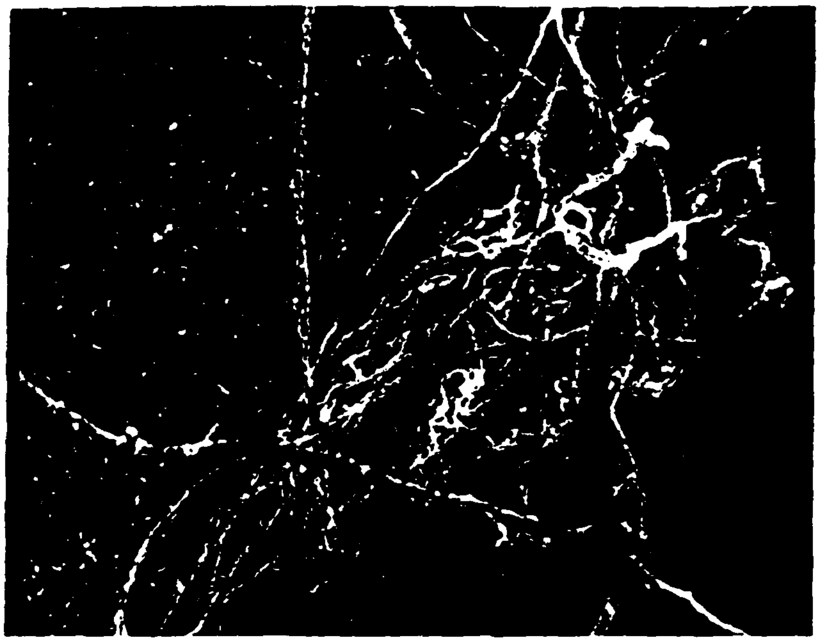
THUS, IT MIGHT BE POSTULATED THAT THE FIBERS ON THE ENGINE FILTERS COULD BE FROM THE COTTON IN THE FUEL TRUCK FILTER. HOWEVER, SINCE THE TRUCK FILTER HAD A HIGH PERCENTAGE OF FIBERGLASS, AND IF THE TRUCK FILTER DID INDEED BREAK UP, WHY DON'T WE SEE MORE SILICA ON THE ENGINE FILTERS? ONE MIGHT ALSO SPECULATE THAT THE SILICA WHICH IS MORE DENSE THAN THE COTTON SETTLES TO THE BOTTOM OF THE TANK AND DOES NOT GET PUMPED OUT AS DO THE COTTON FIBERS.

Date : 3/26/85

Analyst : PAV/CCB

Approved by: JAM

MML# 05357
SAMPLE 23910-2



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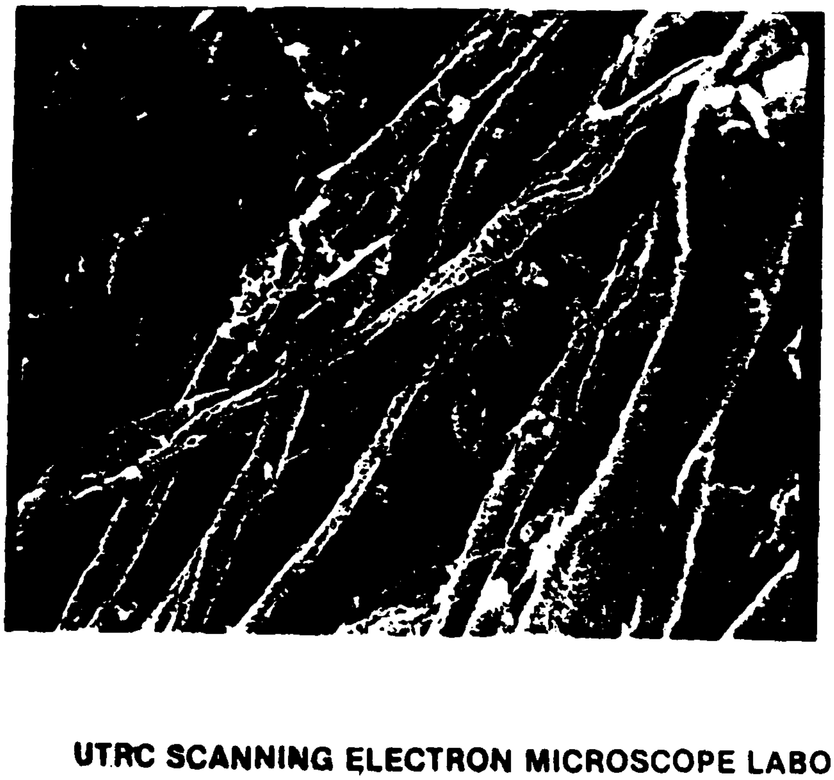

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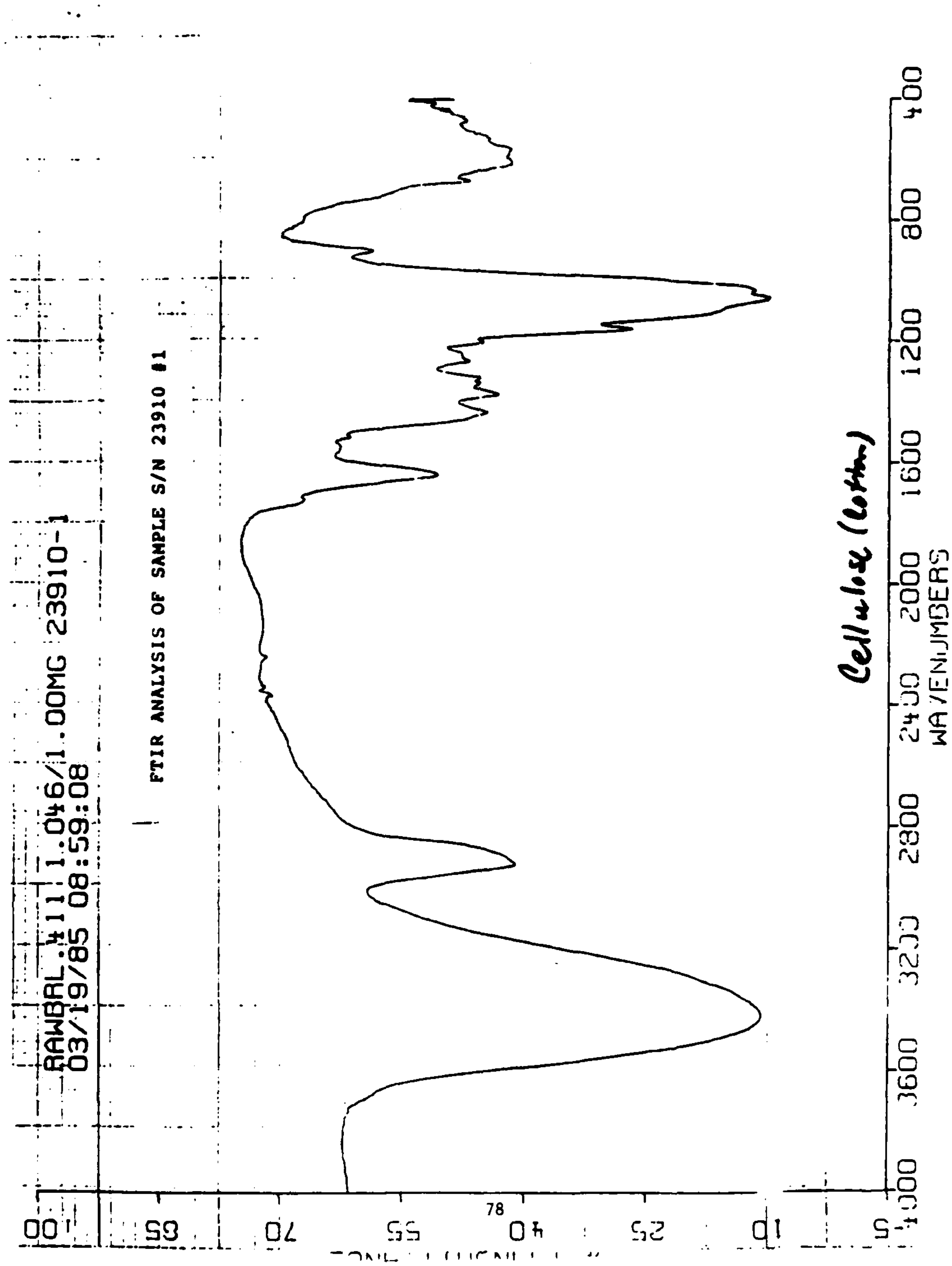
UTRC SCANNING ELECTRON MICROSCOPE LABORATORY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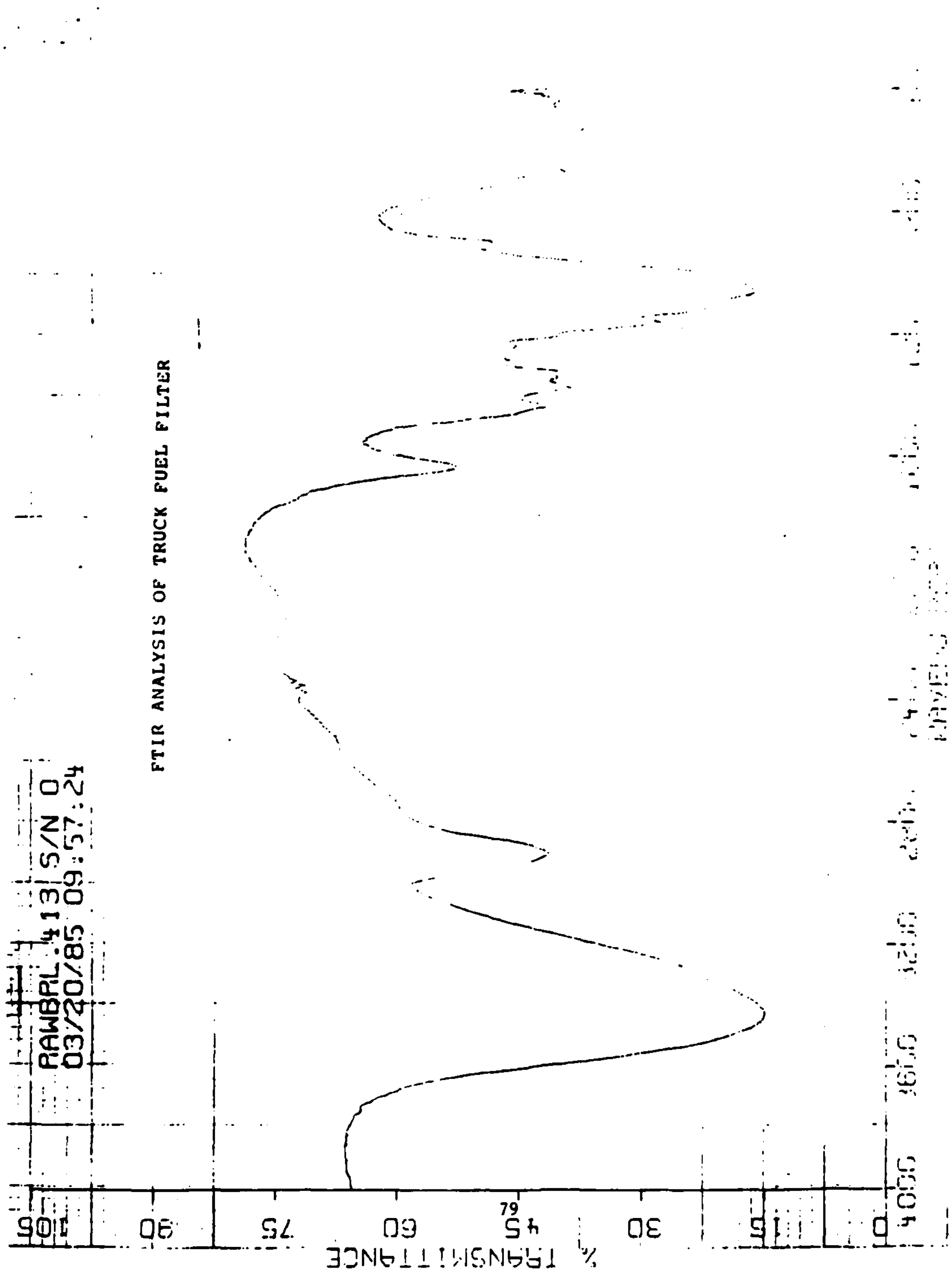
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90
75
60
45
30
15
0
4000
3600
3200

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AMXSN-UK-SE (LTC NICHOLS) 1
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AMXRO-EG (DR MANN) 1
AMXRO-CB (DR GHIRARDELLI) 1
P O BOX 12211
RSCH TRIANGLE PARK NC 27709-2211

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NEW CUMBERLAND PA 17070

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VAIHINGEN, GE
APO NY 09128

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PETROLEUM ACTIVITY
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BLDG 247, DEFENSE DEPOT TRACY
TRACY CA 95376

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CENTER
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AMXST-BA 1
FEDERAL BLDG
CHARLOTTESVILLE VA 22901

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ST LOUIS MO 63120

TRADOC LIAISON OFFICE
ATTN: ATFE-LO-AV 1
4300 GOODFELLOW BLVD
ST LOUIS MO 63120

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FORT STORY VA 23459

HQ
US ARMY TRAINING & DOCTRINE CMD
ATTN: ATCD-SL-5 (MAJ JONES) 1
FORT MONROE VA 23651-5000

DIRECTOR
US ARMY RSCH & TECH LAB
(AVSCOM)
PROPULSION LABORATORY
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CLEVELAND OH 44135-3127

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US ARMY NATICK RES & DEV LAB
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STRNA-U 1
NATICK MA 01760-5000

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FORT KNOX KY 40121

CDR
101ST AIRBORNE DIV (AASLT)
ATTN: AFZB-KE-J 1
AFSB-KE-DMMC 1
FORT CAMPBELL KY 42223

CDR
US ARMY WESTERN COMMAND
ATTN: APLG-TR 1
FORT SCHAFTER HI 96858

CDR
US ARMY LOGISTICS CTR
ATTN: ATCL-MS (MR A MARSHALL) 1
FORT LEE VA 23801-6000

PROJECT MANAGER
PETROLEUM & WATER SYSTEMS
ATTN: AMCPM-PWS 1
4300 GOODFELLOW BLVD
ST LOUIS MO 63120

CDR
US ARMY AVIATION CTR & FT RUCKER
ATTN: ATZQ-DI 1
FORT RUCKER AL 36362

CDR
US ARMY ARMOR & ENGINEER BOARD
ATTN: ATZK-AE-AR 1
ATZK-AE-LT 1
FORT KNOX KY 40121

CDR
6TH MATERIEL MANAGEMENT CENTER
19TH SUPPORT BRIGADE 1
APO SAN FRANCISCO 96212-0172

CHIEF, U.S. ARMY LOGISTICS
ASSISTANCE OFFICE, FORSCOM
ATTN: AMXLA-FO (MR PITTMAN) 1
FT MCPHERSON GA 30330

CDR
US ARMY SAFETY CENTER
ATTN: PESC-SSD (MR BUCHAN) 1
FORT RUCKER AL 36362

DEPARTMENT OF THE NAVY

CDR
NAVAL AIR PROPULSION CENTER
ATTN: PE-33 (MR D'ORAZIO) 1
P O BOX 7176
TRENTON NJ 06828

CDR
DAVID TAYLOR NAVAL SHIP R&D CTR
ATTN: CODE 2830 (MR BOSMAJIAN) 1
ANNAPOLIS MD 21402

JOINT OIL ANALYSIS PROGRAM -
TECHNICAL SUPPORT CTR 1
BLDG 780
NAVAL AIR STATION
PENSACOLA FL 32508

CDR
NAVAL AIR SYSTEMS CMD
ATTN: CODE 53645 (MR MEARNS) 1
WASHINGTON DC 20361

CDR
NAVAL RESEARCH LABORATORY
ATTN: CODE 6180 1
WASHINGTON DC 20375

OFFICE OF CHIEF OF NAVAL
RESEARCH
ATTN: ONT-07E (MR ZIEM) 1
ARLINGTON, VA 22217

CHIEF OF NAVAL OPERATIONS
ATTN: OP 413 1
WASHINGTON DC 20350

CDR
NAVY PETROLEUM OFC
ATTN: CODE 43 (MR LONG) 1
CAMERON STATION
ALEXANDRIA VA 22314

DEPARTMENT OF THE AIR FORCE

HQ, USAF
ATTN: LEYSF (COL CUSTER) 1
WASHINGTON DC 20330

HQ AIR FORCE SYSTEMS CMD
ATTN: AFSC/DLF (MAJ VONEDA) 1
ANDREWS AFB MD 20334

CDR
US AIR FORCE WRIGHT AERONAUTICAL LAB
ATTN: AFWAL/POSF (MR CHURCHILL) 1
WRIGHT-PATTERSON AFB OH 45433

CDR
SAN ANTONIO AIR LOGISTICS CTR
ATTN: SAALC/SFT (MR MAKRIS) 1
SAALC/MMPRR 1
KELLY AIR FORCE BASE TX 78241

CDR
WARNER ROBINS AIR LOGISTIC CTR
ATTN: WRALC/MMTV (MR GRAHAM) 1
ROBINS AFB GA 31098

CDR
HQ 3RD USAF
ATTN: LGSF (MR PINZOLA) 1
APO NEW YORK 09127

CDR
DET 29
ATTN: SA-ALC/SFM 1
CAMERON STATION
ALEXANDRIA VA 22314

OTHER GOVERNMENT AGENCIES

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LEWIS RESEARCH CENTER
MAIL STOP 5420
(ATTN: MR. GROBMAN) 1
CLEVELAND OH 44135

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
VEHICLE SYSTEMS AND ALTERNATE FUELS PROJECT OFFICE
ATTN: MR CLARK 1
LEWIS RESEARCH CENTER
CLEVELAND OH 44135

DEPARTMENT OF TRANSPORTATION
FEDERAL AVIATION ADMINISTRATION
ATTN: AWS-110 1
800 INDEPENDENCE AVE, SW
WASHINGTON DC 20590

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